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BOND STRENGTHS AT THE COMPOSITE ENAMEL INTERFACE.

A COMPARATIVE STUDY BETWEEN NORMAL AND

FLUOROSED ENAMEL.

A thesis submitted to the University of Sydney as partial requirement for the degree of Master of Dental Science (1982).

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INTRODUCTION

Composite resin restorative material and the acid etch technique team up to provide the most effective aesthetic anterior tooth repair known to modern dentistry. It is now possible to achieve that which practitioners have sought since the earliest beginning of dentistry. The new tooth coloured restorative materials actually bond to the tooth structure and are strong enough to withstand the forces of mastication.

An 'acid etching mystique' has developed since Buonocore (1955) first suggested that the improved retention resulting from etching could be useful in many restorative procedures.

Out of this concept, a subculture has emerged, made up of those who splint mobile teeth, attach pontics directly to abutment teeth, seal pits and fissures and even glue teeth back in place.

The use of acid-etching for bonding of the composites has suggested many changes. The scope of these new techniques is limited only by our imagination, ingenuity and discretion.

Dr. Buonocore's (1955) original premise, a technique of etching enamel to improve retention has finally achieved prominence. This procedure is reversible, economical, can be repeated, if necessary, with minimal cost and discomfort to the patient and destruction (or removal) of tooth structure.

In recent years, studies have evaluated the ability of the acid-etch technique to improve both the adaptation and retention of the composite resins to tooth structure. Variable results have been reported for the bonding of cervical lesion restorative materials to enamel and dentine. However, much of the research has been limited to normal enamel and the fluoride content of the enamel has been disregarded. Hence, it was the purpose of this investigation to compare the adhesive bond strengths and penetrativity of resin into normal and fluorosed enamel and to evaluate the role fluorosis plays, if any, as far as the retention of composites to etched enamel is concerned.
CHAPTER I
STRUCTURAL ASPECTS OF ENAMEL

1.1 Gross Appearance

The crown of the tooth is covered by the hardest tissue of the body, the dental enamel. This consists of an acellular calcified material of epithelial origins made up of 92-96 percent inorganic matter, 1-2 percent organic material and 3-4 percent water by weight (Johansen E., 1964).

Enamel is thickest in the region of the cuspal prominences and thinnest at the base of deep pits and fissures and at the cervical region of the crown. This enamel may be pitted and/or discoloured due to intrinsic developmental anomalies - such as hypoplasia or fluorosis (Shafer, W.G., Hine, M.K. and Levy, B.M., 1969). Hypoplasia may also be exhibited as a number of white or brown spots, lines or diffuse zones in a number of locations throughout the crown (Kostlan, J. and Pluckova, M.).

With increasing age, enamel tends to show increasing numbers of cracks. This may be due to repeated exposure to temperature variations and to decrease in permeability because of secondary dentine formation.

1.2 Chemical Composition

Organic: The organic fraction constitutes approximately one percent of the enamel. Its exact chemical composition remains unclear, but analyses indicate that it is made up of proteins, peptides, insoluble protein and citric acid (Stack, M.V., 1954). The soluble protein represents the fraction of the enamel protein which goes into solution when enamel is
demineralised with ethyl diamine tetra acetic acid or weak organic acids. Both soluble and insoluble proteins are present in about equal amounts (Scott and Symons, 1979).

**Inorganic:** The principal inorganic component of enamel is apatite. This mineral comprising approximately 95 percent of enamel is mainly calcium hydroxyapatite \( \text{Ca}_10(\text{PO}_4)_6(\text{OH})_2 \). In areas where individuals receive fluoridated water, topical fluoride therapy or fluoride supplement, the hydroxyl ions may be replaced in part to form fluorapatite. This fluorapatite is less soluble than hydroxyapatite (Mjor and Pindborg, 1973, Bloom and Fawcett, 1975). Several other elements also exist in trace amounts of which sodium, magnesium and carbonate appear relatively more abundant.

**Water:** Water constitutes approximately 4 percent of the enamel. It is present both in a loosely bound, or free, form and a firmly bound state, in a 4:1 ratio. The loosely bound, readily replaceable water is easily removed even at room temperature and is that water which is lost during air drying. The firmly bound water is distributed in the region of prism periphery (Gwinnett, A.J., 1966).

This layer does not seem to influence adhesion appreciably. This is probably due to the fact that the adhesive, itself, depending on its chemical composition, may dissolve or displace most, if not all, of this water when it is brought into contact with the adherend surfaces (Huntsburger, J.R., 1966).
1.3 Microscopic Appearance

Light microscopy has demonstrated that human enamel consists of approximately twelve million units called prisms or rods. In cross-section, the prism consists of a central core or head region and a peripheral or tail region which is an extension of the core between the head regions of adjacent prisms to form interprismatic material. This prism extension should not be confused with what has been previously identified as interprismatic 'cementing substance' (Poole and Brooks, 1961; Sicher, 1966). The new prism configuration has come to be referred to as a keyhole structure of enamel. The border of the keyhole shaped prism is manifest by light microscopy as a narrow, dark line less than one micron in width. The dark boundary represents a refractive phenomenon due to light dispersion at the region between prisms. This border is often referred to as the prism outline. In longitudinal section, prism outlines are seen as a number of relatively parallel refractive lines establishing the prism periphery. In contrast, the cores appear translucent and homogenous.

The prisms do not run a straight course from the amelo dentinal junction out to the enamel surface. In the inner two thirds of enamel, there is considerable decussation or crossing of groups of prisms so that in longitudinal section some may appear to be cut transversely, while neighbouring groups appear to be cut obliquely. This contributes to an optical phenomenon called Hunter-Schreger bands. Not all prisms reach the surface (Innes and Shroff, 1966; Buonocore, 1975; Scott et al, 1974; Osborn and Ten Cate, 1976) from which may be concluded that some odontoblasts die before the full thickness of organic enamel matrix is deposited.
These rods or prisms are narrowest, about 3 microns in cross-section, near the dentino enamel junction gradually increasing in size to about 6 microns at the enamel surface (Sicher, H., 1966).

The structure and course of enamel is best described by Osborn (1968b) as a series of one prism thick, interlocking, open ended tapered cones, the bases of which constitute the surface enamel and the open ends of which lie on the dentino-enamel junction. Within each cone, the prisms wave from side to side, the bends of the prisms in one cone all being the same and slightly out of phase with the bends of the prisms in adjacent cones.

At the ultramicroscopic level of examination, the enamel is seen to be composed of a multitude of long crystallites measured between 20-30 nanometres in width and 150 nanometres in length (Buonocore, 1975). In the central part of the heads, the hydroxyapatite crystallites lie predominantly parallel to the prism axis, whereas in the peripheral regions and in the tail, the crystallites are inclined away from the long axis of the prisms (Arends, 1973; Hardwick et al, 1965).

Enamel sectioned along the prism axis and across the head-tail direction shows the crystallites of the tail region as cross-cut and those of the head region showing their long axis. However, if enamel is sectioned along the prism axis but parallel to the head-tail direction, the crystallites are depicted as running along the prism axis at the upper edge of the prism and dipping down towards the tail throughout the rest of the prism (Meckel et al, 1965a and 1965b; Griebstein and Helmcke, 1965; Romanuik and Shroff, 1965; Scott et al, 1974; Buonocore, 1975). These sudden changes in crystallite orientation at the boundaries between
FIGURE 1  a) Prism arrangement and subdivision of the keyhole in head and tail

b) Course of prisms
prisms serve to define the cross-sectional keyhole shape of the individual prisms.

1.4 Fluorosed Enamel

Dental fluorosis is a term used to describe or refer to that defect manifested on human enamel caused by fluoride intoxication during the period of teeth development. Mjor and Pindborg, (1973, p. 108) recommended the use of the term 'Enamel Fluorosis' or 'Endemic Dental Fluorosis' to refer to this same condition because it usually occurs in well defined geographical areas.

Clinically, dental fluorosis is characterised by faint white flecking of the enamel, white patches or striations or in more severe cases by yellow, black or brown stains and hypoplasia of varying degrees. On account of roughness and porosity of the enamel surface, secondary staining from food pigments is likely to be superimposed.

In severe fluorosis, there may also be confluent pitting with the teeth appearing corroded, and loss of tooth form. Sometimes the enamel chips off easily, because of its brittleness (Forrest, J.R., 1965).

The ultrastructure of fluorosed human enamel is, amongst other things, characterised by an increased porosity, particularly of the outer half to two-thirds of the enamel, positioned deep to a relatively well mineralised surface layer.

Under an electron microscope, fluorosed enamel is characterised by periprismatic gaps or channels almost entirely lacking in enamel crystals
(Fejerskov, Johnson and Silverstone, 1974; Sandstrom et al, 1978; Awazawa, 1962). The individual crystals usually are regular hexagons in cross-sectional profile, often with large intercrystalline spaces, especially at the prism periphery and in the tail region. The size and gross organisation of prisms do not appear to differ from that of normal adult enamel; (Fejerskov, Johnson and Silverstone, 1974) only the orientation of the crystals seems to exhibit a gradual change.
CHAPTER II
THE ACID ETCH TECHNIQUE - BONDING TECHNOLOGY

2.1 Retrospections on Bonding

The acid etch technique was developed and introduced by Buonocore in 1955. With this simple, yet innovative method of etching enamel with phosphoric acid, resin restorative material could be strongly bonded to enamel without the need for the excessive removal of tooth structure that is frequently required in more conventional restorative techniques.

Enamel is predominantly hydroxyapatite, \( \text{Ca}_10(\text{PO}_4)_6(\text{OH})_2 \). The hydroxyapatite crystals are packed together to form prisms which, in the mature tissue, are flattened hexagons in transverse section. The prisms are oriented at right angles to the enamel surface, thus providing a lattice work surface that when selectively etched produces an ideal base for the mechanical attachment of resins.

Acid etching of the enamel is therefore, currently being used either to retain the material or to improve marginal adaptation or to achieve both improved retention and marginal adaptation.

The nature of the retention achieved by acid etching enamel has usually been considered to be a simple mechanical interlocking by resin into pores created in the enamel surface. The term 'adhesion' has frequently been applied to the state in which the material is retained to the etched surface. This seems to be inappropriate since true adhesion is associated with interfacial bonding through molecular attraction - a phenomenon which is considered to play a negligible role in the retention
of composite resin to an acid etched surface. 'Bonding' seems to be a more appropriate term since it describes an observable fact, yet does not describe the mechanism for attachment, such as whether it is due to the physical or chemical forces of adhesion and mechanical retention in undercuts, pores, capillaries or crevices.

2.2 Scientific Basis of Bonding Technology

Lee and Swartz (1970) described three basic criteria for bonding. The surface with which bonding is to occur should be a) similar to the subsurface, b) free of contamination and c) smooth and uniform.

These criteria reflect both chemical and physical considerations - wetting, surface free energy, contact angle and surface tension. Does enamel meet these criteria under clinical considerations?

In the mouth, the enamel surface is constantly undergoing changes as a result of reactions involving deposition, removal and redeposition of organic and inorganic substances to attain the lowest possible energy state. This reaction involves essentially the same physical and chemical forces of molecular attraction responsible for establishing adhesion. As a result of such changes, tooth surfaces in the natural state tend to be more or less fully reacted and are not suitable as bonding substrates for dental resins.

To improve the bonding qualities of enamel, an acid etching technique is used to remove a few microns of outer surface to expose underlying enamel. The acid etching also creates microscopic and submicroscopic pores and crevices in the underlying enamel. Thus, after it is washed and dried, the etched enamel offers a large, high energy, porous surface that renders
the surface wettable and therefore, more conducive to bonding (Glantz et al., 1980). This high energy state must be preserved. Salivary contamination (Hormati, A.A., 1980) and the application of fluorides (Sheykholeslam, Z., 1972) at this time should be avoided as these alter the surface energy and significantly reduce the bond strength to enamel (Sheykholeslam, 1972).

With presently available restorative materials, bonding to enamel appears to be best achieved in the dry condition. If maintained in a relatively dry state, the bonding could last indefinitely. When an appropriate restorative material is applied to a dry, etched enamel surface, it is likely to be attracted to it by physical and/or chemical forces of adhesion to establish molecular contact. These same forces of adhesion are also instrumental in filling the microspaces in etched enamel by capillary action. After hardening, the multitude of projections of resin material that have penetrated into the microscopic capillaries, voids and crevices also serve to mechanically retain the resin in the enamel surface. It is not known, however, how long the forces of adhesion between enamel and restorative material can resist eventual displacement by water. The adhesive may nevertheless be firmly retained mechanically in enamel by tags or projections of adhesive into the capillaries, pores and crevices created by acid etching (Powell, 1975).

The importance the regularity, consistency and even distribution of etch patterns have on bond strength and the durability of restorations clinically has not been clearly established. It has been shown that while bond strengths are greater with 30 to 50 percent acid concentration, the difference between these values and those obtained on surfaces etched with 10 to 70 percent acid were not as great as one might anticipate, based on
differences in the depths of the etch and appearance of the etched surface (Young, 1975). Most of these bonding tests have been performed soon after preparation of the in vitro samples and usually without stressing by thermal cycling. Hence, such results may not be indicative of the effect on bond strength of long term immersions under oral moisture conditions.

2.3 Role of Fluorides in Bonding Techniques

Considerable misunderstanding seems to exist regarding the role of topical fluorides in bonding procedures. Many instances have arisen when the use of prophylaxis pastes containing fluorides or topical fluoride treatments prior to etching have been questioned.

Lee et al. (1972) found that when enamel was treated with a fluoride solution prior to etching, little or no bonding was obtained. Hoffman et al. (1969), who had the same opinion, added further that enamel pre-treatment with sodium or stannous fluorides reduced the etching action of phosphoric acid, probably because of a decrease in enamel crystallite solubility.

In contrast, Rowland et al. (1979) claimed that there is virtually no evidence that fluoride incorporated in enamel prior to etching will significantly interfere with etching or bond strengths. De Paola (1971) showed that fluoride acquired from the acidulated topical fluoride solution is poorly retained and easily removed under oral conditions in a short time.
Another area of controversy is the use of fluorides as part of the etching solution or immediately prior to regular bonding. Studies show that fluoride application after etching produced reaction products or precipitates, namely $\text{Ca}_5(\text{PO}_4)_2 \cdot \text{F}$ and $\text{CaF}_2$ from sodium fluoride treatments and $\text{Sn}_3(\text{PO}_4)_2 \cdot 3\text{F}_3$ and $\text{CaF}_2$ from stannous fluoride treatments (Kochavi et al, 1975; Nordquist et al, 1975). The reaction products seem to interfere with optimal adhesive penetration resulting in weaker bonds and/or bonds that will not survive as long under conditions of oral moisture (Sheykholeslam et al, 1972; Gwinnett et al, 1972).

In quite marked contrast, studies by Chow and Brown (1974) and Takahashi (1977) involving the application of fluoride solutions to etched enamel, found that there was no interference with sealant penetration and Low et al (1976) showed that the treatment of etched enamel with 8 percent stannous fluoride solution prior to resin application significantly increased the tensile bond strength.

Hence, there seems to be no agreement as to the use of fluoride solutions either prior to or after etching enamel. Perhaps the most important use of fluoride is after bonding procedures of all types. During etching, considerably more enamel surface usually is intentionally or unintentionally etched than is subsequently covered by adhesive, such as interproximal areas etched by acid spillover. Etched enamel is highly reactive and readily combines with and better retains many times more fluoride than a natural unetched enamel surface. This large amount of fluoride, thus acquired by etched but uncovered enamel from a topical fluoride application may render the enamel more resistant to cavities.
2.4 Importance of Bonding Agents

Another area of confusion relating to the acid etch technique is whether an intermediate layer of low viscous, non composite resin between composite restorative and etched enamel is beneficial.

Buonocore et al (1968) and McLundie and Messer (1975), suggested that improved surface penetration would be possible with a resin of reduced viscosity. However, other authors have considered the unfilled resin to be unnecessary (Jorgensen, 1975a; Pahlavan et al, 1976). In fact, opinions are about equally divided regarding the need for a low viscosity bonding agent prior to application of the restorative material (Barnes, 1977; De Paola et al, 1971; Dogon, 1975; Fan et al, 1975; Forsten, 1977; Low et al, 1976; Meurman and Nevaste, 1975; Nordenvall, 1978; Qvist et al, 1977; Rider, 1977a; Roberts, 1978). Most of these comparative studies have been done in vitro and conclusions based on microleakage, tag amounts or lengths and less frequently on the basis of bond strengths.

Microleakage studies as well as bond tests are usually performed soon after preparation of the in vitro samples and usually without stressing by thermal cycling. Hence, such results may not be indicative of the effect on bond strength of long term immersion under oral moisture conditions. Besides, scanning electron microscopy of tag length and amounts may not readily show existing differences in amounts of interconnecting microspaces among the tags produced by the use of a bonding agent as compared to those produced by applying a much more viscous restorative material directly over etched enamel surface. The existence of differences in amounts of interconnecting microspaces at the interface between enamel and restorative material could eventually spell differences
in microleakage patterns and in the clinical survival of a bonded restoration. These can only be assessed by long term clinical observation (Buonocore, 1981).

Hence, Buonocore feels that the use of a low viscosity bonding agent over etched enamel prior to the application of a restorative material has long term clinical advantage. Support for this idea comes from work of Rider (1977b) who additionally tested samples after thermal cycling to better simulate certain oral conditions. The use of bonding agents resulted in higher bond strengths after thermal cycling. It was concluded from the results that while the use of bonding agents may not be improving initial in vitro bond strengths, they might provide for a more durable bond in the clinical situation. This, however, is contrary to a study by Ortiz et al (1976) who said that thermal cycling tended to increase the microleakage and decrease the bond strength. Additional evidence of the long term benefits of the use of bonding agents is available from the one year study of Golpayegani (1978), who showed that low viscosity primers increased tensile bond strength, reduced microleakage and produced longer, denser, more numerous tags.
CHAPTER III
THE ACID ETCH TECHNIQUE – ETCHING TECHNOLOGY

3.1 Pretreating Enamel to Increase Adhesion

Chemical treatment of teeth that produces a modified surface is a good method for the improvement of adhesion between tooth structure and dental restorative materials. In 1955, Buonocore reported that treatment of enamel with 85 percent phosphoric acid increased adhesion of enamel to acrylic filling materials. He suggested that increased adhesion may be due to such factors as –

1) A tremendous increase in surface area caused by the etching action of the acid.
2) The exposing of organic framework of the enamel that serves as a network in and about which acrylic can adhere.
3) The formation of a new surface due to precipitation of new substances such as calcium oxalate or an organic tungstate complex, to which the acrylic might adhere.
4) The removal of an old fully reacted enamel surface, exposing a fresh, reactive surface that would be more favourable for adhesion.
5) Producing the presence on the enamel surface of an absorbed layer of highly polar phosphate groups derived from acid used.

At that time he was not sure of the mechanism involved but felt the acid treatment could be clinically and biologically safe.

Since that time, several investigators have looked into 'the acid etch technique' to study more precisely how the acid could serve to increase
the bonding of resins and now the composites.

In addition to the work of Buonocore, Silverstone and Gwinnett must be mentioned who have contributed to the understanding of the pretreatment of enamel as an aid to increasing the retention of plastic restorative materials. From these studies we now know,

1) Acids dissolve the axial portion of the prism heads so that the etched outer surfaces perpendicular to the direction of prisms have a 'honey-combed' appearance and etched surfaces parallel to the prism show troughs and ridges which would be evident in cut or fractured enamel surfaces.

2) Microscopic pores are created in the enamel by a surface active agent creating an extensive surface area for bonding as well as spaces and openings into which an adhesive resin or composite can flow and polymerise.

3) Mechanical retention probably plays a dominant role in the bonding process as irregular peaks and valleys are revealed in the etched surface, in scanning electron microscope studies.

4) Surface roughness is created which contributes to the shear strength of an adhesive, or resin which is being bonded to the tooth.

5) By pretreating the tooth surface with a low pH surface active agent - (phosphoric acid), the usual hydrophobic low energy tooth surface can be converted to a more wettable, hydrophilic high energy tooth surface. The low energy adhesive resins will tend to wet the high energy surface more readily.

6) Enhanced bonding to acid conditioned surfaces is associated with the presence of 'prism like tags' of the adhesive cement or restorative materials apparently formed by the penetration of the material into enamel spaces created by the acid. Penetration is said to be greatest at the interprismatic area.
3.2 The Effect of Acid Solution on Enamel

The application of an etchant, usually phosphoric acid, to the enamel surface causes a reaction with the inorganic calcium salts (Smith et al., 1976). As a result, the enamel surface loses contour relative to adjacent unetched enamel. A 30 percent solution of phosphoric acid produces a surface loss of 10 microns and a depth of histologic change of 20 microns, for a total depth effect of 30 microns. At the base of the etched region, the enamel is modified by the etchant and rendered 'porous' due to partial loss of minerals (Silverstone, 1974).

Numerous studies have investigated the effect of acid solutions on enamel (Poole and Brooks, 1961; Poole and Johnson, 1967; Sharpe, 1967; Gwinnett, 1977; Johnson et al., 1971; Nichol et al., 1973; Jongebloed et al., 1973; Retief, 1973b; Silverstone, 1975). These studies indicate that when acid solutions are applied to enamel surfaces, a selective dissolution of the large inorganic component occurs at sites related to the cores and prism peripheries. The initial reaction of the acid attack is the opening up of a narrow gap in the prism boundary zone (Johnson et al., 1971; Simmelink, 1974). The attack could then follow several directions, either towards the prism cores or along and around the prism boundaries. The primary direction of attack, however, is independent of the etchant used (Gwinnett, 1971; Silverstone, 1975).

Acid etching produces a variety of topographic patterns which reflects differences in solubility due to micromorphologic and chemical variations in enamel. Silverstone et al. (1975) described three basic types of surface characteristics in etched enamel.
In the most common, called type 1 etching pattern, prism core material was preferentially removed, leaving the prism peripheries relatively intact. In the second, type 2 etching pattern, the reverse pattern was observed. The peripheral regions of prisms were removed preferentially leaving prism cores relatively unaffected. In the type 3 etching pattern, there was a more random pattern, areas of which corresponded to type 1 and 2 damage together with regions in which the patterns of etching could not be related to prism morphology. All three etching patterns were found in single samples of etched enamel, suggesting that there is no one specific etching pattern produced in human dental enamel by the actions of acid solutions.

Poole and Johnson (1967) suggested that the different etching patterns produced with either an acid or a chelating agent, were due to difference in orientation of crystallites relative to the direction of attack, together with differences in chemical composition between central and peripheral parts of enamel prisms. Little is known about the chemical differences between these areas, but the role of crystallite orientation seems to be significant.

Enamel crystallites dissolve in acid more quickly along their c-axis than in a direction perpendicular to this axis (Johnson, 1966; Sharp, 1967; Boyde, 1971). Hence, when the attack is parallel to the prisms, as is the case on the enamel surface, the heads or centres of the prisms are preferentially dissolved, because in this region the crystallites lie parallel to the prisms. On the other hand, when the acid attack is perpendicular to the long axis of the prisms, as is often the case with enamel cavity walls, the tails or borders of the prisms are more reactive and the heads are left moderately intact, because the crystallites in the centre of the prisms are
also oriented perpendicular to the acid attack and would not dissolve that rapidly (Sharp, 1967).

However, the acid does not always follow the same regular patterns of dissolution. Variations are found from site to site on the same tooth and also between teeth and teeth from different individuals as well as at different ages (Arakwa, 1979; Galil and Wright, 1979). Various explanations for this directional reactivity have been suggested.

Arends (1973) related this to the presence of dislocations in the central regions of some enamel crystallites, which he called enamel lattice imperfections. These dislocations are regions of undersaturation and hence are most susceptible to acid attack.

Johnson et al (1971), Nichol et al (1973) and Scott et al (1974) suggested that higher organic contents at prism junctions could account for the reduced solubility in acid, because the crystal surfaces are protected. Prism junctions are sites where there is an abrupt change in crystallite orientation and where more intercrystalline space exists which could be filled with hydrated organic matrix.

The slow accumulation of trace elements, for example, fluorine, at sites adjacent to diffusion pathways hinder the movement of ions and causes variations in the dissolution pattern (Poole and Johnson, 1967). As a result, clinically, it has been found necessary to increase the etching time.

Johnson et al (1971) suggested, as one further reason for the differences in etching patterns produced by either acids or chelators, that the difference in size and electric charge exhibited by the different
demineralising radicals might play a part. In contrast, Silverstone et al (1975) showed that there is no one specific etching pattern produced by acid action on human dental enamel suggesting that differences in etching patterns were independent of the acid or chelator used.

The spread of the attack, following initiation, could also be aided by the molecular sieve behaviour of enamel (Poole et al, 1967). Enamel is a porous tissue that contains approximately 0.1 to 0.2 percent by volume of space (Gwinnett, A.J., 1966). Many of the pores communicate to allow for transport of tissue fluid (Bergman, 1963) and ions in solution (Hardwick, 1961). Acid etching enhances the size of these pores as well as increases their depth up to 20 to 30 microns from the tissue surface.

3.3 The Effect of Acid Solution on Dentine

An acid solution, when applied to cut dentine, results in dissolution and partial demineralisation. Microscopically, this effect can be described as follows.

First, the surface layer of smeared tissue caused by the cutting instrument is disorganised and removed. Second, a superficial layer of the highly organic intertubular dentine is partially demineralised, so that a smooth fibrous pannus covers the cavity walls and floor (Hoppenbrouwers et al, 1974). Thirdly, the highly calcified walls of the dentinal tubules are more completely dissolved. Where the tubules are cut in cross-section, their exposed entrances are widened and funnel downwards to a depth of about 12 μm (Lee et al, 1973; Gwinnett, 1973; Brannstrom, 1974).
Clinically, this opening and widening of the tubules has two effects. First, they allow a ready pathway by which toxic materials can reach the pulp, and second, they cause an increase in the wetness of the cavity floor, thus reducing the possibility of achieving an adhesive restorative system (Barnes and Kidd, 1980a).

3.4 Etching Agent and Duration of Application

Buonocore (1955) initially used 85 percent phosphoric acid for his etch of enamel. This strength was apparently chosen in deference to the industrial use of phosphoric acid used in treating metal surfaces to obtain improved adhesion of paint and resin coatings.

Phosphoric acid, the present etching agent of choice, is available commercially in either solution or gel. A number of different concentrations, ranging from 30 to 65 percent are available. Assuming the same etch time, lower concentrations of acid tend to be more destructive of the enamel than higher concentrations (Gwinnett, 1965). Concentrations of phosphoric acid over 65 percent tend to show minimal changes. Chow and Brown (1973) and Soetopo et al (1978) showed that concentrations below 30 percent were unacceptable because the product from the action of phosphoric acid on enamel was insoluble and would remain as a contaminant on the surface. In concentration above 30 percent, a soluble monocalcium monohydrate salt is formed which can be removed by washing.

The concentrations of acid, producing consistent, more or less evenly distributed relatively deep etch patterns appear to be in the range of 30 to 50 percent (Retief, D.H., 1978).
Silverstone (1974) using a 30 percent unbuffered solution of phosphoric acid showed that a 60 second exposure produced the most consistent and evenly distributed etch over an enamel surface. Silverstone's (1975) later studies confirmed his earlier results, and it has become generally accepted that etching with 30 to 40 percent phosphoric acid will provide the ideal surface contour for retention.

In comparison, Buonocore (1975) who developed the ultraviolet curing nuva system of resins, still feels that 50 percent phosphoric acid buffered with 7 percent zinc oxide, as used with that system, is optimal. He feels that the histologic depth of the etch is less important than the number and width of pores created.

A study by Rock (1974) however, tends to support the use of phosphoric acid in the 30 percent range. When comparing the bond strength of Silverstone's recommended 30 percent phosphoric acid, with the more commonly used 50 percent phosphoric acid buffered with 7 percent zinc oxide, Rock found an increase in the tensile strength of a sealant bond of over 50 percent with weaker acid. However, other studies have reported no significant difference in bond strengths between these two acid concentrations (Retief, 1974a, 1975; Williams et al, 1976).

From the preceding discussion it would be apparent that although phosphoric acid is generally regarded as the best acid for etching, its ideal concentration is disputed. There is undoubtedly an ideal depth of etching which should be deep enough to provide resin tags of adequate length. However, if the tags become too long they may fracture when subjected to stress, resulting in a loss of retention. Besides, exaggerated etching could weaken tooth structure between the tags causing
the enamel itself to fracture under stress (Phillips, 1976).

The etched surface is of critical importance to the strength of the bond, for it is into this roughened surface that the resin flows forming the 'tags' that are the bases for retention. The surface not only must be etched for the correct time with the ideal strength of the best acid but, also it must be dry and free of contaminant when the resin is applied.

Recently, an acid gel, Delphic, Lee Pharmaceuticals, rather than an acid solution has been advocated by some to achieve better bond strengths. Newman (1969) from his study concluded otherwise and said that clinical impression seems not to support any advantage of gel over liquid except that gels offer significantly greater control in acid placement.

3.5 Remineralisation After Acid Etching

The ability of the acid etched enamel to remineralise in saliva has become important with the development of pit and fissure sealants, the use of citric acid cavity cleansers and the etching of walls of cavity preparations to be restored with composite resins. If these techniques were to be used successfully, the immediate and long term damage caused to enamel surface must be assessed. If the etched enamel surfaces that are not covered by the dental material are able to return to a 'normal' condition within a reasonable period of time, then the acid etch technique may not be harmful (Albert and Grenoble, 1971).

Albert and Grenoble (1971) and Arana (1974) showed that in vivo remineralisation of acid etched enamel takes place rapidly and that remineralisation is complete within 96 hours. Muhlemann et al (1964)
showed that the characteristic prism end patterns of acid etched enamel disappeared when exposed to the oral environment for 2-48 hours. Silverstone (1977) found the solubility rate of acid etched enamel returned to normal after 48 hours of exposure to oral fluids indicating uptake of both mineral and organic components from saliva.

These findings, however, conflict with the findings of other workers. Wei and Koulourides (1972) and Pickel et al (1975) showed that acid etched enamel does not exhibit complete remineralisation nor, under in vivo conditions, regain the original surface hardness. Meurman and Asikainen (1976) showed that acid etched enamel exposed to the oral environment gained only 84 percent of its original hardness. McDougall and Adkins (1966) showed that after remineralisation, the enamel surface never returned to original optical appearance - some slight loss of birefringence always persisted indicating inability of etched enamel to completely remineralise.

Observations using the scanning electron microscope indicate that enamel mineralisation following acid etching is achieved by the filling of etched microspaces with salivary deposits which become calcified into globular substances (Meurman and Asikainen, 1976; Meurman and Mikko, 1976). This rehardened enamel surface, however, does not exhibit the same solid, non-porous, intact structure. Histologically, some definite defects and micropores could be detected.

In summary, therefore, it can be concluded that though, clinically, the etched enamel appears to return to normal within 48-96 hours and the solubility rate of this remineralised enamel equals that of the adjacent unetched enamel within 48 hours, microscopically and histologically, it's
never the same as the original again. Hence, the unnecessary acid etching of intact enamel by acid solutions used in the acid etch technique should be avoided (Meurman and Asikainen, 1976; Meurman and Mikko, 1976). Further, since it has been shown (Koulourides et al., 1961; Pigman et al., 1964; Hoffman et al., 1969; Gron and Amdur, 1975) that fluorides play a definite role in the remineralisation phenomenon, application of fluoride solution after completion of the restoration would not cause any harm.
CHAPTER IV

THE ACID ETCH TECHNIQUE - PENETRATION STUDIES

Maximum retention and resistance to microleakage can be obtained only if composite resin adapts optimally to etched enamel. The quality of the resin tags produced under different circumstances has been assessed in several ways including direct vision with optical and scanning electron microscopes, shear and tensile tests and microleakage studies.

When the resin is placed on the etched enamel surface, it spreads across the surface and enters the enlarged enamel micropores (Gwinnett, 1965). The rate at which this occurs depends upon the rheologic properties of the resin including its spreading pressure and penetration coefficients (Fan et al, 1975). It has been shown that penetration coefficient, which in turn depends upon viscosity, may influence the degree to which the resin penetrates into enamel (Buonocore et al, 1968; Dogon, 1976). This is contrary to studies of Asmussen (1977c), Jorgensen and Shimokobe (1975) who found no differences in the adaptation of high and low viscous resins to etched enamel.

When dental composites are applied to etched enamel, it is only the resin monomer that enters the enamel micropores, as the filler particles are too large. This resin encapsulates the enamel crystallites (Gwinnett et al, 1967) as well as enters into the crystallites themselves (Simmelink et al, 1974). This ultramicroscopic relationship may be regarded as a 'composite' in which the crystallites are the filler and the resin the matrix.

Some resins do not require an intermediate layer of sealant. These, so called 'wet film' composites, contain sufficient free monomer to form
retentive tags similar in length to those formed when intermediate sealants are used (Asmussen, 1977c). Laboratory data have shown an enhancement of tensile bond strength for certain composites when using an intermediate layer (Buonocore, 1968). Other studies contest the value of an intermediate bonding resin of low viscosity (Retief, 1981). It has yet to be shown whether there is any clinical significance to such observations.

Adhesiveness to pretreated enamel is possibly influenced by the homogeneity of the mix and the cohesiveness of the composite material itself (Tani and Ida, 1978). Hence, for optimal penetration into the tissue, resin placement should be achieved clinically before the resin begins to polymerise. Further, the composite should be mechanically mixed for better results, since hand mixing tends to incorporate air bubbles in the mix. The method of placement is equally important (Jacobsen, 1976). A fine tipped, soft bristle brush is recommended for routine application of acid, using a dabbing or light stroking motion. The soft bristles prevent a heavy rubbing or scrubbing mode of acid application that would result in decreased retention due to the fracture of interstitial enamel surrounding the micropores.

The enamel should be etched until it exhibits a matt, dull, whitish or lightly frosted appearance (Buonocore, 1981). An enamel surface so characterised provides a sufficient amount of indepth spaces for retentive tag formation along with thick, sturdy appearing prism remnants in and about which the resin can harden to produce a strong attachment.

The formation of resin 'tags' first described by Gwinnett and Buonocore (1965) can be used as an index of resin penetration into the tissues.
Measurements of tag lengths vary from 5 to 50 microns (Gwinnett et al., 1967; Gwinnett et al., 1973; Pahlavan et al., 1976; Retief, 1973; Silverstone et al., 1975) depending upon the area of the enamel etched and the type and concentration of etchant used. The enamel of teeth from areas where there is endemic fluorosis has increased resistance to acid etching (Abramovich, 1973). The same is true of the prismless enamel (Sheykholeslam and Buonocore, 1972). Doubling the etching time in the primary dentition in which prismless enamel is most common, or lightly grinding the surface (Conniff et al., 1976) will expose the prismatic enamel below the surface. This facilitates greater resin penetration and enhances retention of the resin.

Resin penetration into enamel rods exposed longitudinally, as in incisal angle fractures, is also minimal (Gwinnett, 1973). To facilitate greater penetration of resin in such cases, beveling the margin of preparation is recommended. This increases the surface area of the prism rods enhancing etching.

After etching for the proper time using the appropriate method with acid of optimal concentration, the area should be subjected to copious water lavage for 15 to 30 seconds. Soetopo and his coworkers (1978) recommended a 60 seconds wash as compared to 15 seconds for improved results. Washing removes the side product surface deposit of soluble calcium salts from the treated enamel surface. Failure to do so may well inhibit effective resin penetration.
CHAPTER V

THE ACID ETCH TECHNIQUE - SOME CLINICAL CONSIDERATIONS

5.1 Introduction

Initially, the acid etch technique was used purely as a means of bonding resins into the occlusal pits and fissures to prevent caries. Soon it became evident, through the research of Buonocore, that another ideal area for use of the system was with the restoration of anterior fractures.

At the present time there is hardly an area of dentistry, excepting perhaps exodontics, where the acid etch technique cannot be applied. Some of the most significant uses are listed in the following sections.

5.2 Pit and Fissure Sealing

The term sealant is used to describe a resin material that is introduced into the occlusal pits and fissures of caries susceptible teeth, thus forming a mechanical physical protective layer against the action of caries producing bacteria and substrates.

The introduction of such materials has provided the missing link for the prevention of dental caries. In the past, despite diligent oral hygiene, optimal fluoride environment and adequate diet, occlusal caries was unavoidable for most people. The means to prevent occlusal caries is now in the form of pit and fissure sealants.

The simple technique, unlike Hyatt's (1923) prophylactic odontotomy and Bodecker's (1964) enamel fissure eradication, does not involve removal
of any healthy tooth structure. The liquid resin is applied onto acid treated occlusal enamel surfaces. The resin flows into the fissures and the etched micropores, forming intimate contact with the enamel and conferring a high degree of resistance to caries by covering the susceptible fissures (Buonocore, 1970; Lee and Swartz, 1971; Rock, 1973).

5.3 Preventive Resin Restoration

The preservation of healthy tooth structure by conservative preparation technique is an irrefutably desirable pursuit. Acid etched resins can be used to prevent secondary caries attack without having to resort to any further removal of tooth structure. This has been called the preventive resin restoration and leads to the preservation of a great amount of healthy tooth structure, which would otherwise normally be removed in the name of extension for prevention (Simonsen, 1978).

Basically this technique consists of removing only carious tissue. No extension for prevention is carried out. Instead resins are allowed to flow into adjacent caries susceptible areas to seal them from the oral environment thus preventing further destruction from caries. Teeth are treated according to the size of preparation necessary to remove caries. Depending upon the size and location of preparation, either filled, unfilled or both filled and unfilled resins may be used.

5.4 Incisal Fracture Restorations

A clinically proven technique for the restoration of incisal fractures involves the successive steps of preparation, pulp protection, acid etching, bonding, composite insertion and finish and glazing (Hinding, 1973).
The most important aspect is the preparation. Resin penetration into enamel rods exposed longitudinally, as often happens in incisal angle fractures, is only about 5 microns (Gwinnett, 1973). To facilitate greater penetration of resin in such cases, it has been recommended to bevel the margin of the preparations (Jordan et al., 1977). The bevel or chamfer shoulder cuts diagonally across the rods to increase surface area and has the additional benefit of improving margin aesthetics of the restorations. Black et al. (1981) on the other hand showed that the fracture of the featheredge incisal edge restorations were significantly lower than those of restorations placed on bevelled preparations. However, the retentive forces of restorations in each of the two procedures were of the same order of magnitude. The ease of performing the clinical procedure may therefore be of major consideration.

5.5 Vital Bleaching

The vital bleaching technique is successfully applicable to teeth discoloured through intrinsic enamel staining and certain cases of tetracycline involvement. A typical example of intrinsic enamel staining that is amenable to vital bleaching treatment is the dark brown discolouration frequently observed in fluorosed teeth.

The labial surface of the teeth is etched by phosphoric acid for one minute, followed by washing and drying. Bleaching solution, a mixture of one part ethyl ether and five parts 30 percent hydrogen peroxide is applied to the etched enamel. Ether lowers the surface tension and permits optimal penetration of bleaching solution into etched enamel.
5.6 Additional Uses

In addition to the above uses, resin technology can be used as an intermediate technique for cases where cost factor is an important consideration. Such a procedure would be useful for space maintenance (Swaine and Wright, 1976), traumatic or periodontal splinting (Andreasen, 1972; Rochette, 1973) fixed prosthesis repair (Scheer and Silverstone, 1975) and repair of porcelain work.

In all the above restoration preparations, the enamel must not be removed from the teeth since etched dentine will not provide as strong a bond as etched enamel (Gwinnett, 1977). No preparation of the abutment teeth is indicated when using a composite to attach a fixed restoration. In the upper arch, case selection is limited to patients where the existing occlusion will accommodate a thin casting without creating occlusal interferences. Occlusion is seldom a factor in lower arch case selection. Application of this restorative technique to posterior problems is not recommended due to occlusal loads.

In conclusion, therefore, the composite restorative material and the acid etch technique originally used as a pit and fissure sealant has now drawn the attention of other specialties of dentistry and enforced into the treatment armamentarium of orthodontists, periodontists and prosthodontists. However, the technique must be used with careful attention to details and proper case selection.
CHAPTER VI

COMPOSITE RESTORATIVE MATERIAL - BASIC COMPONENTS

6.1 Introduction

The term 'composite' as discussed by Dominich V. Rosoto (1969) is, 
"... a system that is created by the synthetic assembly of two or more 
materials; namely, a compatible resin binder to obtain specific 
characteristics and properties. They differ in composition or form on 
a macro scale. Components do not dissolve or otherwise merge completely 
into each other although they act in concert. Normally the components 
can be physically identified along with the interface between components. 
The behaviour and properties of this interface also generally control 
the properties of the composite".

In 1956, the attention of the dental profession was focussed on the 
possible use of this industrial concept of reinforced 'composite' 
materials for dental restorations. Subsequent work at the U.S. National 
Bureau of Standards led to the development and patenting of a composite 
material consisting of silane coated silica filler in a bis-GMA resin 
binder (Bowen, 1963). The basic components of the composite system are 
given below (Jacobsen, 1981).
TABLE I  Composite Restorative Materials - Detailed Components

<table>
<thead>
<tr>
<th>Function</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base monomer</td>
<td>Difunctional acrylate (eg bis-GMA)</td>
</tr>
<tr>
<td>Diluent monomer</td>
<td>Methacrylic acid, MMA, EGDM</td>
</tr>
<tr>
<td>Reinforcing filler</td>
<td>Quartz, boro-silicate, barium glass</td>
</tr>
<tr>
<td>Silane coupling agent</td>
<td>Organo-silane</td>
</tr>
<tr>
<td>Polymerisation inhibitor</td>
<td>Hydroquinone</td>
</tr>
<tr>
<td>Polymerisation catalyst</td>
<td>Benzoyl peroxide</td>
</tr>
<tr>
<td>Polymerisation initiator</td>
<td>Tertiary amine or photo-initiator</td>
</tr>
<tr>
<td>Plasticiser/catalyst vehicle</td>
<td>Dibutyl phthalate</td>
</tr>
<tr>
<td>UV absorber</td>
<td>Organo-benzophenone</td>
</tr>
</tbody>
</table>

6.2 Resin Matrix

Most commercial composites are based on bis-GMA or similar difunctional methacrylate monomers. Bis-GMA due to its higher molecular weight and pseudo-cross-linking properties provides lower setting shrinkage, higher strength and low water absorption. However, it is a viscous sticky material and requires dilution to make it usable. For this purpose viscosity controllers in the form of methacrylic acid or methylmethacrylate monomer are added. This dilution alters the physical properties of the material, lowering the strength and increasing the water absorption.

6.3 Filler Particles

Bowen chose powdered silica glass as the filling agent for his initial composite resin. He determined that it was possible to incorporate a high
percentage of filler and yet retain a workable resin mix if a bimodal system was employed. In such a system two particle sizes of fillers are used. The filler particles are coated with a coupling agent (Vinyl Silane), so that they might adhere to the resin matrix (Bowen, 1964).

The type and nature of the filler particles vary from brand to brand and include quartz, glass beads and ceramic particles. The filler is usually present in the proportion of about 75 percent by weight (Jones et al., 1977), and the range of particle size is about 3 to 75μm.

The recently introduced microfine composites have a filler content of 33 to 50 percent by weight, with the greatest portion being colloidal silica with submicroscopic particles ranging from 0.007 to 0.14μm.

The shape of the filler particles may be of clinical significance. Smooth round particles pull more readily from the resin matrix than do the irregularly shaped particles (Valcke, 1978). The greater retention of irregularly shaped particles may be advantageous either for mechanical interlocking or for better bonding of filler particles to the resin matrix via the coupling agent.

6.4 Radio-opacity

Modern composite resins are radio-opaque to some degree. This is a function of the filler particles. Salts of heavy metals such as barium are added during the preparation of the ceramics or glasses from which the fillers are made. The radio-opacities of a wide range of composites have been measured by Abou Tabl et al. (1979). Different brands show different degrees of opacities.
6.5 Unfilled Resin

Unfilled resin, sometimes termed 'bonding agent' is supplied in the form of a viscous fluid, dispensed as base and catalyst. The unfilled resin polymerises completely only when air is excluded by the placement of the supervening composite resin.

The use of unfilled resin as an intermediate layer between the etched enamel and composite resin has been proposed for two reasons. First, the fluid resin is supposed to flow more readily into etched enamel and secondly, the resin will not readily polymerise until it is covered by the composite filling. Its use will allow several cavities to be filled with the same mix of composite. However, neither of these suppositions can be accepted uncritically (Chapter II, 2.4).

6.6 Chemical Combinations

**Paste/Paste System** (Concise, Adaptic, Cosmic)
Both pastes contain the monomer and filler but differ in that one contains peroxide and the other amine. On mixing, the peroxide-amine reaction initiates polymerisation. This system has the advantage that the ratio of monomer to filler remains constant irrespective of the proportions in which the two pastes are dispensed. Thus the physical properties of the set material will not be affected by slight discrepancies in proportioning.

**Paste/Liquid Systems** (Smile, Perfit)
The paste contains monomer, filler and amine. The liquid contains the peroxide activator. Accurate proportioning is difficult and inconvenient. Wet mixes may result.
Powder/Liquid Systems (Posile, HL72)

The powder contains the filler and peroxide. The liquid contains the monomer phase and the amine phase. Variations in powder/liquid ratio have profound effects on the properties of the set composite. Hence, not a popular product among the profession.
SCOPE OF INVESTIGATION

Composite resin restorative material and the acid etch technique team up to provide the most effective anterior tooth repair known to modern dentistry. Yet it is not exactly the ideal filling material of choice, for an ideal filling material would be truly adhesive, have the same physical properties as teeth, be tooth coloured and biologically compatible with the pulp.

From the review of the literature it would become clear that in order to achieve this 'ideal concept' continuous research is being done to improve the material and technique of application. Along with this, controversies concerning the use of an etchant, the etching time, the type of etching solution and whether low viscosity resin would give better results especially in relation to the bond strengths and adaptation are continuously being evaluated.

It was the purpose of this research to investigate the adaptation of 'concise composite resin' material into etched fluorosed enamel, compare it with that of normal enamel and evaluate it in relationship to past researches.

This investigation was carried out in two parts. Part A examined the bond strengths. Part B examined the etched enamel surface available for bonding using a scanning electron microscope.
CHAPTER VII

METHOD A

An investigation of the Bond Strengths at composite enamel interface. A comparative study between normal and fluorosed enamel.

7.1 Pilot Studies

A number of pilot studies were performed before the actual experiment was conducted to determine a variety of factors. Initially, the acrylic cylinder with the tooth embedded was secured to the chuck. The preparation of composite specimens and its attachment to the etched enamel was similar to that used in the final procedure. The only difference was that the 'donut-shaped' dies and consequently the replicas were much smaller in dimensions (Figure 2). Once the composite had set, it was allowed to stand for three hours, a hole drilled across, a string passed through it and attached to one end of the tensilometer, the other end having been occupied by the chuck. On pulling the assembly apart, the composite fractured at a point of weakness, the hole. Hence, the method was modified. This is described in section 7.3.

Using this method, five specimens were subjected to tensile strength tests after allowing a setting time of three hours.

The results of the study are recorded in Table II. The table gives the mean value and standard deviation of the tensile force required to separate the resin material from the etched enamel surface.
TABLE II Tensile force required to break the resin enamel bonds.
Setting time 3 hours. Etching time 1 minute.

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Tensile Bond Strength in MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.21</td>
</tr>
<tr>
<td>2</td>
<td>5.32</td>
</tr>
<tr>
<td>3</td>
<td>5.35</td>
</tr>
<tr>
<td>4</td>
<td>5.67</td>
</tr>
<tr>
<td>5</td>
<td>5.67</td>
</tr>
<tr>
<td>Mean ± S.D.</td>
<td>5.84 ± 0.70</td>
</tr>
</tbody>
</table>

During the investigation, there was an occasional cohesive or partial cohesive break for the material used. This could possibly be the result of poor mixing technique, poor packing technique, air bubbles in the material or incomplete polymerisation of the material itself.

To assess the polymerisation time a similar experiment was conducted allowing the composite to set for seven days before it was subjected to tensile force. The results of the five specimens tested are tabulated in Table III.
FIGURE 2 Donut-shaped dies used in the initial study

FIGURE 3 Metal die used to prepare specimens for diametrical tensile strength
FIGURE 4  Donut-shaped die modified and enlarged for final investigation
TABLE III Tensile force required to break the resin enamel bond.
Setting time 7 days. Etching time 1 minute.

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Tensile Bond Strength in MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>16.68</td>
</tr>
<tr>
<td>2</td>
<td>19.83</td>
</tr>
<tr>
<td>3</td>
<td>19.00</td>
</tr>
<tr>
<td>4</td>
<td>17.35</td>
</tr>
<tr>
<td>5</td>
<td>16.68</td>
</tr>
<tr>
<td>Mean ± S.D.</td>
<td>17.91 ± 1.28</td>
</tr>
</tbody>
</table>

The results indicate a mean tensile force of 17.91 MPa which is much higher than that exhibited by specimens tested after allowing a three hour setting time. Hence, it was decided to carry out the final investigation after allowing the composite to set for seven days.

To prove the validity of the technique and assess the results, it was further necessary to verify the tensile strength of the material used with those of other workers. A comparison was made between the results obtained by packing composite in a metal die (Figure 3) and that packed in the prepared silicon dies (Figure 7). This was necessary to assess the packing technique, the mixing technique and the flow of the composite into the die itself.
A comparison was also made of the diametrical tensile strength of composite specimens prepared by the silicon die technique and the tensile strength required to pull the material apart.

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Tensile Strength of Composite in MPa</th>
<th>Metal Die</th>
<th>Silicon Die#</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>41.45</td>
<td>57.28</td>
<td>22.35</td>
</tr>
<tr>
<td>2</td>
<td>39.71</td>
<td>50.67</td>
<td>23.92</td>
</tr>
<tr>
<td>3</td>
<td>42.30</td>
<td>50.00</td>
<td>21.09</td>
</tr>
</tbody>
</table>

Mean ± S.D. 41.15 ± 1.32 52.65 ± 3.29

Mean ± S.D. 22.45 ± 1.16

Dimensions of Specimens:
*metal die: radius 2.0 ± 0.1mm x height 8mm
#silicon die: radius 2.0 ± 0.1mm x height 8mm

TABLE IV: Comparison of diametrical tensile and straight tensile strength of composite setting time 7 days.
GRAPH 1 Comparison of diametrical tensile and straight tensile strength of composite. Setting time 7 days.
The results of the above study give a mean diametrical tensile strength of 41.15 MPa when composite is packed in metal dies. This agrees with figures given by other workers (Short et al., 1976; Silverstone, 1974). Hence, the mixing and packing technique seem to be adequate. The higher values given by specimens prepared in the silicon dies could be attributed to the uneven thickness of the composite cylinder along its length. Measurement of specimens prepared by this method showed a discrepancy in thickness of up to 0.1mm at different levels. This difference in thickness is expected due to the elastic properties of the rubber impression material. Therefore, the values of the diametrical tensile strength of composite obtained by packing the material into silicon dies can be regarded as within normal limits and the packing technique adequate.

The table also gives the relationship of the bond strengths obtained by subjecting the material to different forces. Diametrical tensile strength gave approximately twice the values as that obtained by pulling the specimens apart. Hence, the tensile bond strength values at the enamel/resin interface should not exceed 25MPa. If the bond is stronger than this force, there would be a cohesive fracture of the composite itself.

From the foregoing, it can be concluded that the technique used to measure the bond strength should give satisfactory results. The mixing of the composite resin, the packing technique as well as the polymerisation time should not give any error in the final investigation. Further, if the enamel composite interface were to fracture, the bond strength values should not be greater than 25MPa.
7.2 Final Investigation

7.2.1 Materials used in the investigation

Teeth:
A total of 30 teeth, 15 normal and 15 fluorosed, were used in the investigation. The normal teeth were all premolars extracted for orthodontic reasons. Immediately after extraction, the teeth were cleansed in hydrogen peroxide, any hard deposits removed with a scaler and then lightly polished with pumice and water. Next, they were examined for damage, if any, due to forceps extraction, selected and stored in physiologic saline solution at room temperature until required.

The fluorosed teeth* came from inhabitants of Muranga, Kiambu and Limuru—all situated around Nairobi, in Kenya. Most of the teeth were molars or premolars extracted because of periodontal reasons or because of caries. No attempt was made to collect teeth from any particular age groups. Neither was the concentration of fluoride in water taken into account because of unavailability of information regarding the same. The only criterion for selection was that the teeth had at least one intact surface free of caries and hypoplastic pits. The teeth were collected immediately after extraction and stored at room temperature in neutral buffered formalin. Formalin was used since it is a good preservative for human specimens.

About a hundred of these specimens were cleansed of all the debris, scraped of any attached calculus and polished with a slurry of pumice and water. Glycerine was avoided because it might interfere with the etching procedure.

*Teeth collected by Dr. D.P. Patel, Dental clinic, Kenyatta National Hospital, Nairobi, Kenya.
From the above sample, fifteen specimens were carefully selected so that each tooth
- exhibited at least one intact smooth enamel surface
- had enough dentine to provide adequate support to the enamel
- was either mildly or moderately fluorosed.

The extent of fluorosis was judged according to Dean's (1934) classification of mottled enamel diagnosis; mild fluorosed enamel exhibited white opacities but did not involve more than 50 percent of the surface. Moderate fluorosis showed distinct brown stains and involved all enamel surfaces.

The selected fluorosed specimens were then stored in physiologic saline solution at neutral pH and at room temperature until required.

**Enamel etching solution:**
Concise Etching Liquid*, a 37 percent ortho-phosphoric acid, was used to etch the enamel prior to application of the composite filled and unfilled resins (Figure 5).

**Composite resin:**
Concise composite resin* was supplied in two 16g containers labelled concise paste A and paste B. The low viscosity unfilled resin was supplied in two 2.5g bottles labelled concise Enamel Bond System Resin A and Resin B (Figure 5). When required approximately equal quantities of paste A and paste B or equal number of drops of resin A and B were dispensed and mixed.

*3M Center, St. Paul, Minnesota, U.S.A.
FIGURE 5 Photograph of concise/enamel bond composite resin material
7.2.2 Method of investigation

Thirty teeth, fifteen from each group of normal and fluorosed, were used in the investigation. Each group of fifteen samples was randomly divided into three subgroups of five each. Subgroup A was etched for 1 minute, subgroup B for 2 minutes, and subgroup C for 3 minutes.

Preparation of Specimens

All the selected teeth were sectioned at the cemento enamel junction using a water cooled diamond wafering blade* in isomet low speed saw* rotating at a speed of 1000 revolutions per minute.

Each tooth crown was then placed with the intact enamel surface face down on a sheet of wax and a length of plastic cylinder tube (6mm long x 6mm internal diameter) positioned over it. Caulk Trayresin# was then poured into the tube so that the tooth was mounted into the top of a cast cylinder of acrylic resin in a way similar to that described by Cadwill and Johannessen (1972).

In the preparation of normal enamel specimens, the mesial or distal surfaces were chosen to face down, instead of Buccal or lingual surfaces, because they were less likely to have been cracked during the forceps extraction and also because a flat area would be much easier to prepare on them. In fluorosed specimens, the best possible surface available was chosen.

Each exposed enamel surface was then ground flat using waterproof aluminium oxide paper down to 600 grade. The surface measured approximately 2.5mm by 3.5mm square. By this procedure, approximately 1.0mm of the outer enamel

*Beuiller Ltd, Evanston, Illinois, U.S.A.
#L.D. Caulk, Company, Milford, Delaware, U.S.A.
was removed. The flat exposed enamel surface was then polished using Floran* prophylaxis paste on a bristle brush (Shey and Houpt, 1980) until scratches were removed and a lustrous surface resembling that of enamel was produced (Figure 6).

'Donut-shaped' dies, having a 3mm diameter opening, 6mm thick, were made with silicon base rubber impression material#(Figure 7). These dies were attached to the prepared flat surfaces of the enamel with sticky wax to assure uniform bond areas and restorations, but not before the enamel was etched.

The acid etchant was applied for one, two or three minutes to the prepared enamel surface. A small mini-sponge* soaked in the etchant was gently rubbed against the enamel surface and although no specific attempt was made to etch the enamel of the tooth surface adjacent to the area to be bonded, some etching was occasionally seen to have taken place. Fresh acid was continuously dabbed on the surface to ensure circulation of fresh acid on the enamel surface at all times. After the prescribed etching time, the enamel was sprayed first with water, then with air and water for a total of fifteen seconds and finally dried with an uncontaminated air stream for five seconds (Dogon and Silverstone, 1975). All enamel surfaces were examined for dull, frosty, matt appearance of adequately etched enamel (Buonocore, 1970).

After the silicon dies had been positioned and fixed with sticky wax, the unfilled resin was dispensed, mixed according to the manufacturers' directions and applied with the help of a small brush. The fixing up of

*Floran, Creighton Pharmaceuticals Pty, Ltd, Sydney, Australia
#Optosil, Bayer Leverkusen, W. Germany
+3M Center, St. Paul, Minnesota, U.S.A.
the silicon dies was a very critical procedure and some of the specimens had to be discarded because the dies were either not properly centered or wax had leaked through and affected the etched area. The filled resin was then dispensed, mixed according to the manufacturers' instructions and the 'donut-shaped' dies filled in such a way that no voids or air bubbles were trapped. The viscosity of the mix made it necessary to force the material into the die to ensure the closest possible adaptation with the etched enamel. A small, tightly rolled pellet of cotton wool held in a pair of tweezers was used to force the first portion of the mixed resin into the die. The die was then slightly overfilled, the whole assembly positioned on the plaster block which was previously made for the purpose of achieving a perfect alignment of composite block and tooth surface, a polyester matrix* adapted on the outer surface, and hand pressure applied to simulate clinical technique (Figure 8).

After five minutes, the matrix was removed and the silicon die cut off. The specimens were examined for any excessive material at the composite/enamel junction. If present, this thin 'flash' was removed with a probe and the area smoothened with a fine grade composite finishing disc# containing a Zirconium silicate abrasive. All restorations were finished approximately 15 minutes after the material had set. Figure 6 shows the finished prepared specimen ready for testing.

7.2.3 Experimental regimen

The above prepared samples were stored in 100 percent humidity at room temperature for seven days and then subjected to thermal cycling. This was carried out by alternately immersing a number of gauze bags containing

#3M Center, St. Paul, Minnesota, U.S.A.
FIGURE 6  Left, prepared enamel surface ready for bonding
Right, tooth with bonded specimen ready for testing

FIGURE 7  Silicon dies - negatives made in original donut-shaped dies
the teeth in water baths maintained at temperatures of 4°C and 60°C. The immersion time in each bath was 30 seconds. The teeth were subjected to a total of 100 cycles.

Tensile bond strength was measured using a Hounsfield Tensometer Type W* (Figure 9). The samples were secured in chucks number 17 and 18 (Figure 10). A set of chucks consists of two pairs or four halves. Each set has two numbers. The two half chucks number 17 were assembled on one end and the two half chucks number 18 on the other end. The locking rings were then slipped over the chucks to keep them together (Figure 11). The mercury in the index column was drawn out and the level adjusted up to zero. With the left hand on the chucks, the handle was turned slowly in a clockwise direction until the chuck assembly began to tighten and become rigid. The mercury index was rechecked and adjusted if necessary. Next, additional force was applied to pull the two chucks apart and the reading recorded when the bond fractured. Five readings were taken for each etching time and tensile bond strength calculated. The machine was hand operated and readings taken directly from the scale.

7.2.4 Examination of resin/enamel surfaces using Scanning Electron Microscopy

Fractured surfaces (Figure 12) of the test specimens used to determine the bond strength were examined using the scanning electron microscope. The procedure used was the same as described in the method B (7.3.3). Figure 13 shows the fractured specimen gold coated and ready for examination.

*Tensometer Ltd, 81 Morland Road, Croydon, Australia
FIGURE 8 Specimen alignment assembly

FIGURE 9 Hounsfield Tensometer Type W
FIGURE 10  Chucks and locking rings number 17 and 18

FIGURE 11  Chuck assembly ready for testing
FIGURE 12 Enamel/resin interface after fracture

FIGURE 13 Fractured interface gold coated for SEM examination
METHOD B


7.3 Materials Used in the Investigation

The enamel etching solution and the composite and unfilled resins used were identical to those in the first part of this study (7.2.1)

Teeth:

Twenty four premolars extracted for orthodontic reasons were cleansed as previously described, examined for damage due to forceps extraction, selected and stored in physiologic saline at room temperature until required. Similarly, twenty four fluorosed teeth exhibiting either mild or moderate fluorosis were selected, cleansed, examined and stored until required.

7.3.1 Method of investigation

In this part of the study, enamel surfaces were used to investigate the effects of different etching times. The surfaces examined were transverse and longitudinal sections of teeth and the enamel tooth surface. In each case tooth preparations were carried out using a water cooled tungsten carbide bur* at high speed.

The transverse sections (Figure 14) were prepared by sectioning the crown of the tooth approximately 4mm occlusal to the cemento enamel junction and

*ELA, Germany
FIGURE 14  Diagram illustrating the preparation of a transverse section through a premolar tooth
FIGURE 15 Diagram illustrating the preparation of a longitudinal section through an incisor tooth
FIGURE 16  Diagram illustrating the preparation of a section through an incisor tooth where approximately 2 mm of the surface enamel is removed.
FIGURE 17  Etched normal and fluorosed enamel sections gold coated for SEM examination
a) surface enamel
b) transverse section
c) longitudinal section
d) 2-4mm enamel surface removed
the longitudinal sections (Figure 15) were prepared by sectioning through the crown mesio-distally or Bucco-lingually. In addition to examining the intact enamel tooth surface, the labial surface of the tooth was investigated after removal of approximately 2mm of surface enamel (Figure 16).

Each surface that required etching was etched with 37 percent orthophosphoric acid using a small cotton pellet and a gentle rubbing action. The etchant was removed by washing with water followed by a water and air spray for a total of fifteen seconds; the surface was dried with an uncontaminated air stream for five seconds. During the etching procedure, fresh acid was continuously added to achieve maximum effect.

Each group of transverse, longitudinal and surface enamel specimens were etched for one, two and three minutes respectively to see the etching pattern and behaviour.

7.3.2 Examination of specimens

The prepared specimens were cleaned ultrasonically in distilled water for ten seconds, passed through acetone twice for 5 seconds each and then transferred to trays lined with lint-free tissue paper. After a drying period of 30 minutes (a longer period caused cracks to develop in the specimens), the specimens were mounted onto aluminium stubs using adhesive tape. The specimens were then painted with carbon dag (Acheson Dag)* on the outer surface to facilitate better conduction and then coated with gold to a thickness of approximately 20-40 nanometres in a sputter coating unit# (Figure 17abcd).

*Acheson Dag - Dag 580, Acheson A.N.Z. Pty Ltd, Sydney, Australia
#Dynavac - Dynavac High Vacuum Pty Ltd, Victoria, Australia
The specimens were examined in a scanning electron microscope*, by the mode of secondary electron emission, using accelerated voltages in the range 5 - 15KV. Specimens were examined initially on the television screen for area location and then on the cathode ray tube screen at a scanning speed of 2.5 seconds per frame for final area location and focussing. Photographs were taken at magnifications of x 1000, and x 2000, using the photographic attachment SMU-CS1 with Kodak plus-x pan professional film (s-PxP 120),# at f11 and a scanning speed of 50 seconds per film.

*JSM-U3 - Japan Electron Optics Laboratory Co. Ltd., Tokyo, Japan
#Eastman Kodak Co., Rochester, New York, U.S.A.
CHAPTER VIII
RESULTS

8.1 Method A

An investigation of the bond strengths at composite enamel interface. A comparative study between normal and fluorosed enamel.

8.1.1 Results of final investigation

The results of bond strengths at normal and fluorosed enamel surfaces are shown in Tables V and VI. Table VI gives each value as the mean of five tests carried out using five different specimens. One reading from the group of fluorosed specimens etched for 3 minutes was not included in the mean and standard deviation analysis, because it was very low. This difference in value could be due to faulty technique, possibly malalignment of the specimens while the composite was setting.

Normal Enamel
Mean bond strength at etched normal enamel etched for one minute with buffered 37 percent orthophosphoric acid according to the instructions of the manufacturers was 17.62MPa. The mean values obtained at two minutes and three minutes etching time were 13.85MPa and 10.00MPa respectively. The maximum bond strength was exhibited by specimens etched for one minute, the bond strength decreasing with an increase in etching time. The percentage decrease in bond strength as the etching time was increased from one to two and three minutes was approximately 77% and 56% respectively.
Fluorosed Enamel

Mean bond strength at the fluorosed enamel/composite interface, etched for one minute with 37 percent buffered orthophosphoric acid was 5.73MPa. An etching time of two and three minutes gave mean values of 15.39MPa and 14.95MPa respectively. The maximum bond strength was obtained following an etching time of two minutes, though three minutes etching time did not show a significant difference. The percentage increase in tensile bond strength as the etching time was increased from one to two and three minutes was approximately 267% and 260% respectively.

Hence, the most outstanding findings demonstrated by the testing of tensile bond strengths was the inverse relationship of bond strengths and etching times as related to normal and fluorosed enamel.

8.1.2 Nature of the enamel/resin fracture surface

Scanning electron microscopy of the fracture surfaces of specimens used to determine the bond strengths indicated that fracture normally occurs in the region of the enamel/resin interface. (Figure 18). The enamel fracture surface is similar in structure to the original etched enamel. The reduced detail of the surface can probably be attributed to the presence of residual tags in the enamel surface. Evidence of adhesive failure within the resin was however, not clear. Some specimens did not exhibit any etch patterns but indicated only presence of composite resin material interspread with dark areas, perhaps indicating fracture at the filled/unfilled resin interface or incomplete wetting of enamel surface by the resin during application and adaptation.
TABLE V  Tensile force required to break the resin enamel bond at different etching times.
A comparative study between normal and fluorosed enamel.

<table>
<thead>
<tr>
<th>Specimen Number</th>
<th>Etching Time</th>
<th>Normal Enamel</th>
<th>Fluorosed Enamel</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>14.48</td>
<td>6.30</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>16.68</td>
<td>7.24</td>
</tr>
<tr>
<td>3</td>
<td>1 minute</td>
<td>22.03</td>
<td>5.35</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>17.95</td>
<td>5.35</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>17.95</td>
<td>4.41</td>
</tr>
<tr>
<td>1</td>
<td></td>
<td>13.85</td>
<td>12.59</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>14.79</td>
<td>14.61</td>
</tr>
<tr>
<td>3</td>
<td>2 minutes</td>
<td>13.22</td>
<td>16.69</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>13.22</td>
<td>16.69</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>14.16</td>
<td>16.37</td>
</tr>
<tr>
<td>1</td>
<td></td>
<td>11.33</td>
<td>14.80</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>5.03</td>
<td>14.80</td>
</tr>
<tr>
<td>3</td>
<td>3 minutes</td>
<td>10.38</td>
<td>16.69</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>11.65</td>
<td>5.35</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>11.65</td>
<td>13.54</td>
</tr>
</tbody>
</table>
TABLE VI The mean values and standard deviations of the tensile force required to break the resin/enamel bond - A comparative study between normal and fluorosed enamel.

<table>
<thead>
<tr>
<th>Etching Time In Minutes</th>
<th>Mean Tensile Bond Strengths (MPa)*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Normal Enamel</td>
</tr>
<tr>
<td>1</td>
<td>17.82±2.75</td>
</tr>
<tr>
<td>2</td>
<td>13.85±0.67</td>
</tr>
<tr>
<td>3</td>
<td>10.00±2.83</td>
</tr>
</tbody>
</table>

*Average of 5 specimens ± standard deviation

#Shows average of 4 specimens ± standard deviation
GRAPH II  Mean values and standard deviations of the tensile force required to break the resin/enamel bond - A comparative study between normal and fluorosed enamel
FIGURE 18 Scanning electron micrograph of enamel/resin interface after fracture.

a) Everted edge of composite resin, x 15

b) Surface enamel showing etch pattern and evidence of residual resin, x 500

c) Same area, x 1000

d) No evidence of enamel surface. Fracture surface shows only composite resin, x 500
8.2 Method B


8.2.1 Introduction

Using the method described (7.3.2) etched enamel surfaces of normal and fluorosed teeth were investigated by examining photomicrographs of

i) Normal enamel surface etched for one, two and three minutes

ii) Fluorosed enamel surface etched for one, two and three minutes

iii) Transverse and longitudinal sections of teeth with normal enamel etched for one, two and three minutes

iv) Transverse and longitudinal sections of teeth with fluorosed enamel etched for one, two and three minutes.

Evaluation of the photomicrographs indicate that fluorosed enamel is more resistant to acid etching by phosphoric acid than normal enamel.

A few specimens were examined under the scanning electron microscope after removing about 2 - 4mm of the enamel surface. Comparison of normal and fluorosed enamel etched after reduction of surface enamel did not exhibit any difference in dissolution. The specimens viewed were etched for one minute only. Since there was no apparent difference in the enamel solubility and since such a procedure is rarely used clinically, further investigation of such specimens was not carried out.
8.2.2 Effect of 37 percent phosphoric acid on normal and fluorosed enamel.  

A scanning electron microscopy study

The study showed that the exposure of human dental enamel, either normal or fluorosed, to phosphoric acid solution produced three basic etching patterns.

In the first, called type I etching pattern, there was a generalised roughening of the enamel surface, but with a distinct pattern showing hollowing of prism centres with relatively intact peripheral regions (Figure 19 a). The average diameter of the hollowed regions was approximately 3μm for normal enamel. This was usually somewhat greater than in fluorosed enamel.

In the second, or type II etching pattern, prism peripheries appeared to be removed or heavily damaged. Therefore, the prism cores were left projecting towards the original enamel surface (Figure 19 b; 24 c,d,e). This apparent damage of the peripheral regions of the prisms was seen to extend along the length of the prisms, thus aiding in delineating individual prisms (Figure 20 c,d). When viewed from the original surface (Figure 19 b), separate bundles or columns of materials were seen, the gaps separating them corresponding to the peripheral regions of the prisms. Thus, this type II etching pattern was the reverse of the honeycomb pattern of type I damage, and both patterns were produced by either normal or fluorosed enamel. The type II pattern was found to be the most common of the three patterns observed. This is in contrast to the observations made by Silverstone et al (1975) who found that type I was the most commonly occurring pattern.
FIGURE 19 Variation of enamel etch pattern

a) Type I - preferential dissolution of prism core, x 5000
b) Type II - preferential dissolution of prism periphery, x 5000
c) Type III - no definite etch pattern x 2000
Some etched regions showed neither type I nor type II etching patterns, exclusively. These areas appear as a generalised surface roughening with regions resembling hollowed prism centres adjacent to areas in which the pattern was more consistent with the loss of prism peripheries. In addition, within these areas were regions in which the etching patterns did not apparently conform to prism morphology. These regions were referred to as type III etching patterns (Figure 22 c,d). Sometimes, the whole region in the type III etching pattern was one in which the entire surface topography could not be related to a prism pattern (Figure 19 c).

Longitudinal and transverse sections of etched enamel, when viewed under scanning electron microscope, also showed a similar variation in the etch pattern. Normal or fluorosed enamel etched for one, two or three minutes resulted most commonly in the dissolution of the prism peripheries of the enamel, leaving the prism cores exposed and relatively unaffected (Figure 24 c,d,e; 25 a,b; 26). Less frequently the prism cores were preferentially dissolved leaving raised prism peripheries surrounding the depressed prism cores (Figure 29 a,b). This difference in solubility of the prism components was perhaps caused by the varying angles in which the prisms had been sectioned during preparation before exposure to the acid. The predominant etching pattern on these 'cavity wall' surfaces was similar to the usual etching pattern described above (Type II). Figure 26 c shows the bending effect seen in enamel, taken from a transverse section of a molar tooth. This bending effect, most commonly noticed as the dentino-enamel junction is approached, was due to the variation in prism directions. Some prisms must have been cut and etched in a direction nearly parallel to the prism axis and others in a direction oblique to the axis.
8.2.3 Comparison of etched enamel - normal versus fluorosed

In the above study, a comparison was made between etched normal and fluorosed enamel etched for one, two and three minutes. Every effort was made to observe similar regions and locate similar etch patterns in the specimens for the purpose of evaluation. In longitudinal and transverse section specimens, the photomicrographs were taken from the middle third in an attempt to avoid the bending effect exhibited as the dentino-enamel junction is approached. To get better photographs of some of the regions specimens were tilted and rotated in various directions until a satisfactory view was obtained.

The most outstanding finding of the study was that fluorosed enamel is much more resistant to acid etching by 37 percent phosphoric acid than normal enamel. Further, fluorosed enamel produces the best surface conducive to bonding at three minutes etching interval as opposed to normal enamel which gives a better etch at one and sometimes at two minute intervals.

Etched Surface Enamel, Etched for One, Two and Three Minutes

Photomicrographs 20 to 23 show the effect of etching on normal and fluorosed surface enamel at one, two and three minutes respectively.

Normal enamel at one minute etch (Figure 20 a,b) exhibits a distinct and a definite etch pattern most suitable for bonding, while fluorosed enamel (Figure 20 c,d,e,f) shows only roughening of the surface enamel. Some specimens did exhibit a definite etch pattern (Figure 20 c,d) but the etch was very superficial. Others did not show any enamel dissolution at all (Figure 20 e,f). Only superficial enamel surface reduction was evident.
At two minute etching time, the depth of etch seemed to be equally efficient both in normal (Figure 21 a,b) and fluorosed (Figure 21 c,d,e,f) enamel. The etching pattern, however, was not as clear and definite as that seen in normal enamel etched for one minute. Greater surface roughness seemed to be evident.

At three minutes etching time, the acid attack was clearly evident both in normal and fluorosed enamel. Fluorosed enamel (Figure 22 c,d) however, exhibited a far greater, deeper and a definite etch as compared to normal enamel (Figure 22 a,b). In normal enamel, there was evidence of surface destruction as exhibited by unevenness of the prism periphery in type I patterns (Figure 22 a,b) or selective core dissolution in type II patterns (Figure 23 a,b). This was not observed in fluorosed enamel.

Both normal and fluorosed enamel, however, exhibited selective surface dissolution in certain regions (Figure 23 a,b,c,d). This could be due to difference in compositional elements at different regions.

**Transverse and Longitudinal Sections of Enamel Etched for One, Two and Three Minutes**

Photomicrographs 24 to 30 show the effect of etching by phosphoric acid on transverse and longitudinal sections of normal and fluorosed enamel.

Similar results to that described above are exhibited by these photomicrographs. In brief, normal enamel gives the best, most distinctive and definite etch pattern at one minute etching time (Figures 24 a,b; 27 a,b) while fluorosed enamel gives the same at three minutes etching time (Figures 26 c,d; 30 c,d). In some cases, two minutes etching time
gave equally good results for both normal and fluorosed enamel (Figure 28 a, b, c, d).

Figure 26 a, b show normal enamel in transverse section etched for three minutes. One can easily notice the fragile network of prism peripheries remaining after the effect of the acid. Similar effect is noted in Figure 27 a, b which shows a longitudinal section of normal enamel etched for three minutes. Prism peripheries in this case show evidence of fracture at some places.
FIGURE 20  Surface enamel etched for one minute

a) Normal enamel, x 1000
b) Same area, x 2000
c) Fluorosed enamel, x 1000
d) Same area, x 2000
e) Fluorosed enamel, x 1000
f) Same area, x 2000
FIGURE 21 Surface enamel etched for two minutes

a) Normal enamel, x 1000
b) Same area, x 2000
c) Fluorosed enamel, x 1000
d) Same area, x 2000
e) Fluorosed enamel, x 1000
f) Same area, x 2000
FIGURE 22  Surface enamel etched for three minutes

a) Normal enamel, x 1000
b) Same area, x 2000
c) Fluorosed enamel, x 1000
d) Same area, x 2000
FIGURE 23 Surface enamel etched for three minutes

a) Normal enamel, x 1000
b) Same area, x 2000
c) Normal enamel, x 1000
d) Same area, x 2000
FIGURE 24  Transverse section through enamel, etched for one minute
a) Normal enamel, x 1000
b) Same area, x 2000
c) Fluorosed enamel, x 1000
d) Same area, x 2000
e) Same area, x 5000
FIGURE 25  Transverse section through enamel, etched for two minutes

a) Normal enamel, x 1000
b) Same area, x 2000
c) Fluorosed enamel, x 1000
d) Same area, x 2000
FIGURE 26  Transverse section through enamel, etched for three minutes

a) Normal enamel, x 1000
b) Same area, x 2000
c) Flourosed enamel, x 1000
d) Same area, x 2000
FIGURE 27 Longitudinal section through enamel, etched for one minute

a) Normal enamel, x 1000
b) Same area, x 2000
c) Fluorosed enamel, x 1000
d) Same area, x 2000
e) Fluorosed enamel, x 1000
f) Same area, x 2000
FIGURE 28 Longitudinal section through enamel etched for two minutes

a) Normal enamel, x 1000
b) Same area, x 2000
c) Fluorosed enamel, x 1000
d) Same area, x 2000
FIGURE 29  Longitudinal section through enamel, etched for three minutes

a) Normal enamel, x 1000
b) Same area, x 2000
c) Fluorosed enamel, x 1000
d) Same area, x 2000
FIGURE 30  Longitudinal section through enamel, etched for three minutes

a) Normal enamel, x 1000
b) Same area, x 2000

Fluorosed enamel, x 1000

d) Same area, x 2000
CHAPTER IX

DISCUSSION

9.1 Introduction

Enamel lesions, especially those which require aesthetic restorations are
difficult to treat. The depth of the lesion is one factor, but the
retention of the material is another that determines the success or
failure of the restorations. This has particularly been observed in teeth
exhibiting mild or moderate fluorosis where the acid-etch technique has
not been very successful. This study has confirmed the clinical observation
and suggests that mild or moderately fluorosed teeth should be etched for
at least 2 - 3 minutes to obtain adequate bond strengths as compared to
normal enamel where 1 minute etching time seems to be adequate. Scanning
electron photographs taken to show the depth of etch at different etching
times indicate the same.

Etching is not the only factor affecting the bond strength. Parameters
such as shrinkage during hardening of the adhesive, the mechanical properties
of the adhesive and adherend and the coefficient of thermal expansion of
adhesive and adherend influence the stress concentrations at the interface
as well. In vivo parameters such as moisture and acid produced by bacteria
attack the adhesive bond.

Some of these factors which could be controlled or simulated in the in
vitro technique were considered in the experiment. Others were disregarded
as they involve the manufacture of the material itself.
9.2 Discussion of the Experimental Method

9.2.1 Tooth preparation

All composite resin restorations tested in the bond strength studies were placed directly onto the etched enamel after removal of approximately 1-2\text{mm} of tooth surface. This reduction in enamel surface is bound to affect the tensile bond strength. The greatest surface area for bonding is probably produced when etching occurs on a surface to which most of the prisms are perpendicular. The outer third of the labial enamel probably presents this type of orientation to the highest degree. However, as one approaches the middle and the inner thirds of the enamel, prism orientation becomes considerably less regular with the greatest intertwining and deviation of prism direction from the perpendicular occurring in the inner third of the enamel. The increasing concentration of organic matter as the dentino enamel junction is approached may also be a contributing factor to this reduction.

In our investigation, however, this reduction of enamel surface was necessary in order to obtain a flat surface so that when the specimens are pulled apart, a more accurate reading is obtained.

9.2.2 Enamel prophylaxis

A mechanical cleansing of the enamel is an important first step clinically in the bonding procedure. Miura and his coworkers (1973) showed that maximum bond strengths were developed only when an oral prophylaxis was done before etching. Clearly, acid alone cannot remove all enamel contaminants. This is especially true of calculus, and therefore a careful inspection was made for the presence of this accretion. Because there is
concern for interference of flavouring oils, glycerin and fluorides with the etching process, the use of a watery slurry of flour of pumice was used in the bond strength studies. Bristle brush was used in the cleansing procedure since studies show no qualitative differences between a rubber cup and a bristle brush prophylaxis (Galil, 1975; Taylor et al, 1973).

9.2.3 Etching agent, etching procedure and washing

Concise etching liquid, a 37 percent orthophosphoric acid, was used to etch the filled and unfilled resins. This concentration was selected because the review of literature (3.4) suggests that bond strengths are low at very high and very low acid concentrations rising to a maximum at around 30 - 50 percent (Chow and Brown, 1973). At low and high concentrations less dissolution and more precipitation takes place until at both extremes the enamel remains virtually unchanged. This precipitate must be removed or prevented from forming to obtain optimum adhesion.

Removal of the precipitate can be accomplished by washing. Few studies have been conducted on the question of how long the enamel should be washed immediately upon completion of etching. In a laboratory study, Soetopo and his coworkers (1978) showed a significant increase in bond strength values when enamel was washed for 60 seconds compared to 15 seconds. These observations were made using phosphoric acid in concentrations of 30 percent and lower. In contrast, Williams and Fraunhofer (1977) found a particular combination of etch (60 seconds) and wash (10 seconds) times for optimising bond strengths. The acid used was 50 percent phosphoric acid containing 7 percent zinc oxide.
The washing time of 15 seconds used in this study was based on the compromise between the above two reports and on the suggestions of Dogon and Silverstone (1975) and Gwinnett (1981) who have done considerable work with these etching agents and their effects.

The prevention of the precipitate was accomplished by continually renewing and agitating the acid on the enamel surface during the etching procedure. By this procedure, the concentration of calcium ions is not allowed to rise to the level where precipitation occurs (Beech, 1977).

9.2.4 Thermal cycling

Thermal cycling test carried out in this study used the temperature range of 4°C to 60°C. In previous research (Nelsen et al, 1952; Kopel et al, 1975; Eriksten and Buonocore, 1976a and b; Kidd et al, 1978) specimens were cycled at temperatures separated by a minimum of 50°C within the range of 0°C to 68°C. The values for the temperature range and cycling times used in this study were based on a compromise of the testing regimens of other workers.

Before thermal cycling, the specimens were immersed in water for 7 days. This was done partly to simulate the clinical application and partly to relieve the stresses at the enamel/resin interface. Immersion of the specimens in water would result in water absorption and consequently expansion of the composite. This in turn would compensate for polymerisation shrinkage to a certain extent and reduce stresses at the enamel/resin interface (Martin, 1980).
The temperature range of 4°C to 60°C was greater than that to which restorations might frequently be exposed in the mouth. The immersion period of 30 seconds in each bath was also longer. Such variation in temperatures for such an extended period of time could hardly be tolerated in vivo. Hence, it has been suggested that more realistic temperature limits and cycling time would be 15°C to 45°C and 10 seconds respectively.

9.2.5 Finishing

It has been suggested by many authors (Asmussen, 1975; Mitchem and Granum, 1976; Gjerdet, 1976) that the majority of finishing should be delayed for at least 24 hours in order to permit water absorption which begins 4-6 hours after the insertion of the restoration to close marginal gaps, reduce stresses in the region of the etched enamel/resin interface and to support the enamel margins during finishing.

However, in this study all restorations were finished approximately 15 minutes after the composite resin had set. This method was adapted because the majority of practitioners finish their composite restorations immediately rather than at a subsequent appointment. This could have affected the bond strength at the enamel/resin interface to a minor degree.
9.3 General Discussion of the Bond Strength Studies and Related Factors

Presently, there is no known material capable of forming a permanent adhesive bond with untreated enamel surface under oral conditions. However, satisfactory bonding can be achieved after etching the enamel surface. The findings of the study suggest that mechanical retention plays an important role in the bonding of many adhesives to treated enamel surfaces. The penetration of adhesive material into the microscopic pores in the enamel appears conducive to the retention of the materials. The slight etching of the enamel surface creates a tremendous surface area available for bonding and opens up spaces or pores in the enamel into which the adhesive can flow and ultimately polymerise therein.

Tensile strength of the bond is related to the effective cross-sectional area of all the tags. This may be interpreted as meaning that the length of the tags is unimportant. However, this is fallacious as the strength of the bond is a priori of the energy required to break the bond. The energy to break any one tag \( W = \sigma_b^2 \frac{V}{2E} \), where \( \sigma_b \) is the tensile strength of the tag (hence, resin), \( E \) its elastic modulus and \( V \) its volume; clearly the volume depends upon length as well as cross-section, so the strength (in energy terms) of the bond would be expected to depend upon the tag lengths as well as their effective cross-sectional area.

If the material is strong intrinsically, the strength of the mechanical bond would increase with increased penetration of the material into the enamel. In turn, and assuming that the composite has good 'wetting' properties for enamel, the extent of penetration will depend upon the number of spaces in the enamel into which the material can flow. The depth of the penetration, namely the 'tag length' will also depend upon the speed
of polymerisation of the material. If the material sets rapidly, then any free monomer present in the early stages of polymerisation may have little time to penetrate.

The period in which the permanent tooth has been exposed to the oral environment may also be an important factor governing the degree of etching and hence the amount of spaces created in the enamel. Preliminary observations indicate that teeth which have been in the mouth for only a year or two readily demineralise when treated with the acid solution, while older teeth do not show much evidence of etching. The acquisition of a thick organic cuticle and continued maturation involving changes in surface chemistry of the tooth with age probably protects the surface from dissolution.

The fluoride content of enamel may yet be another factor governing the adaptation of resin to enamel.

Thus a number of factors may empirically be expected to affect the adaptation and bond strengths at the resin enamel interfaces. These are, 1) the efficiency and time of etching procedure 2) the cleanliness and dryness of the surface 3) the viscosity of the resin 4) the mechanical properties of the resin 5) the method of application of the resin to the tooth structure 6) the structure and elemental composition of tooth enamel.

Our investigation was primarily directed towards this last factor, namely the effect the naturally incorporated fluoride has on the bond strength at the resin/enamel interface. The study was designed to test combinations of material, tooth structure and etching. A comparison was made between the etching behaviour of normal and fluorosed enamel and correlated to bond
strength studies.

Using scanning electron microscopy, an attempt was made to study the penetration of resin into etched normal and fluorosed enamel. Preliminary investigation revealed several factors may influence the accuracy with which the resin tags could be measured under a scanning electron microscope. These are -

1) damage during sectioning and acid relief of the sample
2) heat production during the coating of the sample
3) fracture or degradation of resins during the procedures and the variation of the angulation, tilt and height of the pedestal containing the sample in the microscope
4) variations in prism directions.

It was realised that heat production could be reduced in the vacuum evaporator by placing the sample on the outside perimeter of the beam and coating with gold with intermittent pulsations of current. The error in length due to angulation, tilt and height could be corrected to some extent by noting the angle of tilt and by stereomicroscopic examination.

From the preceding paragraphs, it would be clear that accurate measurements of tag lengths would not be possible by scanning electron microscopy alone. Verification of results would be required by preparing another set of specimens and observing them under transmission light microscope and by polarised light. In our study, due to the need to limit the investigation, it was not possible to carry out all the above procedures. Therefore, only etched normal and fluorosed enamel surfaces were observed under scanning electron microscope.
The results of the bond strength studies indicate that normal enamel should be etched for 1-1.5 minutes for maximum retention of composite, while fluorosed enamel require 2.5-3 minutes etching time. Further, 3 minutes etching on normal enamel, results in a substantial decrease in bond strengths.

These results are supported by our studies on etching behaviour and dissolution of normal and fluorosed enamel. Observations using scanning electron microscopy revealed that presence of a significant fluoride content inhibit enamel surface alterations during acid-etching with phosphoric acid. This is in agreement with Hoffman et al, 1969; Lee et al, 1972 and Buonocore, 1975. This inhibition may be due to a variety of factors - 1) presence of trace elements 2) the distribution of organic material 3) the reprecipitation of minerals at prism junctions.

Presence of trace elements

The chief mineral portion of fluorosed enamel in fluorapatite. Fluorapatite and hydroxyapatite conform to the formula Ca$_5$(PO$_4$)$_3$(F,OH). In this formula F and OH can substitute mutually to form a complete series between the essentially pure end members. The species fluorapatite comprises the ranges of composition with fluoride greater than hydroxide and hydroxyapatite has hydroxide greater than fluoride. Its concentration, almost without exception, is present to a high degree in the surface region falling sharply to a low plateau level which continues throughout most of the thickness of enamel. This fluorapatite is less soluble than hydroxyapatite at acid reaction, indicating the need for prolonged etching times as compared to normal enamel. Further, this would explain why we got similar etching behaviour when 1-2mm of enamel surfaces of both normal and fluorosed enamel were removed and etched.
The distribution of organic material

Enamel lesions with dental fluorosis are characterised by periprismatic channels or gaps almost entirely lacking of enamel crystals. Furthermore, the individual crystals usually are regular hexagons in cross-sectional profile, often with large intercrystalline spaces, especially at the prism periphery and in the tail region. These spaces are filled with hydrated organic matrix. The higher organic content of fluorosed enamel could account for the reduced solubility in acid because the crystal surfaces are protected.

The reprecipitation of minerals at prism junctions

When enamel is etched with phosphoric acid, it has been suggested that insoluble complexes may be formed between foreign ions and products of decalcification and that these complexes then form a protective, precipitated layer on the surface under acid attack. Gray and Francis (1963) suggest that this occurs at least in vitro, with the fluoride ions. The accumulation of this precipitate at sites adjacent to diffusion pathways hinders the movement of ions and causes variation in the dissolution pattern. As a result, clinically, it would be necessary to increase the etching time.

Increased etching on normal enamel, on the other hand produces excessive destruction of enamel prisms in all directions. Photomicrographs taken after three minute etching (Figure 23) showed prism periphery dissolution accompanied by a generalised dissolution of enamel leaving small raised 'islands'. Some areas showed fracturing of remaining prism cores. On this evidence, the surface presented after three minute etching would be extremely porous, with many sites available for resin bonding. However,
the application at this surface of mechanical or thermal stress associated either with the placement of the composite resin or with polymerisation contractions during setting, or with different dimensional changes in the tooth and resin from thermal stimuli, would almost inevitably result in the collapse of some of the remaining prisms because of their fragility. These fractured pieces would interfere with resin penetration or prevent resin penetration altogether in certain areas resulting in decreased bond strength.

In our investigation, no attempt was made to evaluate the fluoride content of the enamel of either normal or fluorosed teeth. Neither was the fluoride concentration of the drinking water from where the specimens were obtained available. It would be interesting to know what effect different levels of fluoride content within the enamel has on the bond strengths at the resin/enamel interface.

The fluorosed specimens used in the study were those exhibiting mild or moderate fluorosis, only. Mottled enamel, perhaps, would give much inferior bond strengths due to its increased organic content. Would the acid etch technique be beneficial for restoration of such teeth is yet another question that would be interesting to study?

9.4 Conclusion

The findings of the study suggest that mechanical retention plays an important role in the bonding of adhesives to treated enamel surfaces. The penetration of adhesive material into the microscopic pores in the enamel appear conducive to the retention of the materials. The slight etching of the enamel surface creates a tremendous surface area available
for bonding and opens up spaces or pores in the enamel into which the adhesive can flow and ultimately polymerise therein.

For the etchant studies, teeth were etched with 37% phosphoric acid; the acid was allowed to remain on the surface for the specified time and then rinsed away thoroughly with water. Human enamel, some without a known history of fluoride treatment, and others from an area of known high natural fluoridation were used for the study.

Scanning electron micrographs show that teeth from an area of high natural fluoride are highly resistant to etching with 37% phosphoric acid. It would be interesting to determine the effects of various concentrations of natural fluoride in enamel on the efficacy of the various etching procedures, but due to the necessity to limit the investigation, our study did not include that. In the absence of these data, the present work suggests that in areas of high fluoridation, longer etches with stronger acids are required to achieve adhesion to fluorosed enamel with the resin tested.

Reduction in etching results in some surface effect which affects the wettability and subsequently a decrease in the bond strength of the resin to fluorosed enamel.

Increased etching on normal enamel produces a reverse effect. There is excessive dissolution of enamel in all directions leaving a weak and a fragile framework of prism rods or peripheries projecting between large microspaces. This surface may seem conducive to bonding, but does not give a good bond strength. Application of composite together with other factors (9.3), results in collapse of some of the remaining prisms which affect the penetrativity of the sealants into the micropores and hence the
bond strengths. Therefore etching of normal enamel should be limited to one to one and a half minutes only.

Bond strength studies were conducted on normal and fluorosed human enamel. The teeth used were premolars for normal enamel and molars for fluorosed enamel. Bonds were tested in straight tension and the values were reported as calculated Mega Pascals from the surface area to which the resin actually was applied. After application of the adhesive, all teeth were stored for seven days in water at room temperature and then thermally cycled before being tested. Five specimens were tested for each condition.

The results of the study indicated an inverse relationship between bond strengths and etching times for normal and fluorosed enamel. For normal enamel, best bonding was achieved at one minute etch. Fluorosed enamel gave best results at three minute etch. This confirms our findings of etching studies that reduction in etching and some surface effect that may have been related to wettability resulted in a substantial decrease in the bond strength of the resin to fluorosed enamel.

The standard deviations in the data for bond strengths at normal and fluorosed enamel is very small as compared to other studies. The reason for these low values was not investigated. Slight variations in results, for example, the occasional low bond strength value, may be related to technical errors.

Testing the adhesive joints, in tension, as done in this study, does not necessarily duplicate in vivo forces encountered in service, where components of shear and compression would be much more favourable to mechanical retentive forces than tensile loading.
This work has shown that variations on the time of etching have a definite effect on the interfacial work of fracture. This was supported by evidence of scanning electron microscope. Human teeth from an area of high natural fluoridation were shown to be extremely resistant to etching with phosphoric acid. Such teeth should be etched for longer periods to achieve maximum retention of composite restorations.
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