Influence of an alloy addition on the physical and clinical behaviour of glass ionomer cement.

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Abstract:

These in vitro studies compared the various properties of an experimental high powder liquid content glass ionomer cement (EXPT) with those of a metal addition GIC (Hi-Dense) and disperse phase amalgam (Dispersalloy).

Bi-axial, four point flexural and compressive tests were used to evaluate strength. Six groups of ten specimens were constructed for each test for each material and allowed to set in an oven at 37°C for 60 minutes. Specimens were stored in distilled water at 37°C until testing at one day, one week, one, three, six months and year. It was found that the strength of Hi-Dense increased and then maintained over extended time, whereas the strength of EXPT showed a declined at 3 months.

The bond strengths of the materials to both enamel and dentine were also evaluated. Ten groups of ten teeth, five for each surface for each glass ionomer materials, were prepared. Teeth were aligned leaving the enamel and dentine surfaces exposed. The mixed material was condensed into a cylinder placed on the appropriate surface. These specimens were also stored in distilled water at 37°C. It was found that Hi-Dense had a higher bond strength to enamel that increased with time. The bond strength to dentine was maintained over the test period.

The erosion rate of the materials was evaluated using the lactic acid erosion test. Three groups of six specimens for each material were constructed and tested after one hour, one day and at six months. Each specimen was subjected to an impinging jet of lactic acid solution. The erosion rate was determined by weight loss and dimensional change. It was found that Hi-Dense had a high erosion resistance which was slightly better than the experimental material.

The microleakage, around restorations prepared, using the glass ionomer materials, was evaluated after cyclical loading the restoration-tooth complex. It was found that there was
less leakage around Hi-Dense than EXPT at both the cervical and occlusal margins.

In a clinical comparative evaluation of Hi-Dense with a disperse phase alloy placed as Class I restoration, the indirect assessment showed that Hi-Dense showed greater wear at six months than the amalgam using Ivoclar method of model assessment of wear.

These studies indicate that the incorporation of a metal addition in the glass ionomer may have brought about a slight improvement in some of the properties tested. However, the performance of the experimental material with similar high powder content but no metal addition indicates that the use of a high powder content may be the predominant cause for the possible improvement.
Declaration

I hereby certify that the work embodied in this thesis is the result of my own investigations, unless otherwise stated.
Dedication

This work is dedicated to my dear mother,
brothers, wife and sons who have given me great support.
**Aim:**

The aim of this study is to compare;

* the long term physical properties of a metal reinforced glass ionomer cement (GIC) with those of unreinforced counterpart.

* the strength of glass ionomer cement with amalgam.

* the performance in a clinical trial of the metal reinforced material with amalgam.
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Legend and Abbreviations

- GICs Glass ionomer cements.
- EXPT Experimental glass ionomer cement.
- PAA Polyacrylic acid.
- ADA American dental association.
- Bis-GMA Bisphenol-A glycidyl methacrylate.
- EGDMA Ethylene glycol dimethacrylate.
- TEGDMA Triethylene glycol dimethacrylate.
- UDMA Urethane dimethacrylate
- HEMA Hydroxyethyl methacrylate.
- ACTA Academic Centre for Dentistry Amsterdam.
- VLC Visible light cure.
- DBAs Dentine bonding agents.
- THFMA Tetrahydrofurfuryl methacrylate.
- BS British Standard.
- NHS National Health Service.
- USPHS United States Public Health Service.
- SEM Scanning electron microscopy.
- MPa Mega pascal.
- CRA Clinical Restorative Association.
Chapter (1)

Introduction
1 Introduction:

Dental materials are used to replace tooth tissue that has become damaged or destroyed by a pathological process. Restoration of the tooth to its original form and appearance using a restoration requires some form of tooth preparation. In the process, substantial amounts of sound tooth structure may be removed either to aid retention of the restoration or to provide a greater bulk of restoration giving it greater resistance against the stresses which occur in the mouth. The loss of sound tooth tissue has been reduced in part, following the development of adhesive restorative materials.

Currently there are three distinct types of direct restorative materials. These are amalgam, composite resin and glass ionomer cement (GIC). Amalgam is generally considered to be the material of choice in the posterior segment of the mouth while composite and glass ionomer cement were initially intended for anterior applications. However, they are now considered to be suitable for use in both the anterior and posterior segments. Dental amalgam has a good combination of physical and mechanical properties. It is easy to manipulate and shape to the correct anatomical/occlusal form and its mechanical properties make it resistant to breakdown under occlusal load. However, it has certain limitations. These are:-

1) It corrodes in the mouth (McLean & Gasser 1985).

2) It does not adhere to tooth tissue, therefore mechanical retention is required (Mjör 1985).

3) It has the unattractive aesthetic appearance of metal.

4) It must be placed in sufficient bulk, to avoid brittle fracture. This leads to the removal of sound tooth tissue.
5) Its lack of adhesive properties leading to microleakage.

Although amalgam has proved to be a reliable restorative material, microleakage and secondary caries are major problems (Mjör 1985).

The replacement of amalgam alloys with alternative restorative materials has been the main objective of dental materials' scientists in the last five decades. This is primarily due to concern expressed about the long term biological safety of dental amalgams because of the mercury content in their formulation and the issues regarding the release of mercury into the environment from dental practices and industry (Smart 1981, Hutton & Symon 1986 & Arenholt & Larsen 1996). Another potential disadvantage of amalgam is the amalgam corrosion process which result in the migration of metal atoms into dentine (discolouration of the restored tooth) (McLean & Gasser 1985). The introduction of high-copper alloys has however reduced but not eliminated corrosion.

Attempts to replace amalgam with tooth coloured direct restorative materials have had limited success, with the new materials proving to be less reliable than amalgam when used in the posterior region of the mouth. Composite resins are considered by many to be the main alternative restorative materials for posterior teeth. Composites were originally designed as an enhanced type of filled resin for use in the anterior segment of the mouth. However, the use of optimised fillers, (shape, size and distribution), coupling agents and bonding agents has improved the mechanical and physical properties of composites to the point where a substantial number of practitioners now use them in limited applications in the posterior segments of the mouth. These filled resin systems have relatively low polymerisation shrinkage, a low coefficient of thermal expansion,
high compressive strength and stiffness, radiopacity of a suitable level for diagnostic purpose, an adequate bond to enamel via acid etching and to dentine using dentine bonding agents, and low solubility (Bowen & Marjenhoff 1992). However, to date composites have proved less than ideal suffering from the following limitations;
1) They shrink on polymerisation (Davidson & Gee 1984). This is the primary cause of marginal discrepancy which may lead to microleakage.
2) They are technique sensitive (Pearson 1990). Variations in placement technique can lead to difficulty in manipulation and poor performance.
3) They are more expensive to use than amalgam due to the greater time required to perform the procedure and the requirement for adequate moisture control (Bryant 1991).
4) They are susceptible to failure through secondary caries. Forsten et al (1982) have reported a high level of recurrent caries beneath these restorative materials.
5) They are liable to wear. Bulk wear and inorganic filler loss have both been identified (Phillips et al 1973). The rate of wear is dependent upon filler type, quality of resin (Hirt et al 1984), particle size (Jorgensen 1980) and filler loading together with the quality of filler-matrix interface (Sulong & Aziz 1990).
6) Their thermal properties do not match those of the tooth. This, in combination with the gap resulting from polymerisation shrinkage, can lead to percolation of oral fluid along the interface.

The second alternative to amalgam as a direct restorative is glass ionomer cements (GICs). The invention and development of glass ionomer cement resulted from attempts to improve an earlier aesthetic restorative material, the silicate cement. Initially, Smith (1968) demonstrated that polyacrylic acid and zinc oxide produced a zinc polycarboxylate
cement, which had different properties from zinc phosphate. Wilson and Kent in the early 1970s then used the same acid with a modified silicate glass to produce the prototype glass ionomer cement. This material had similar beneficial properties to silicate cement, in particular it released fluoride over a long period of time. It also has some limitations including a need for early protection from moisture.

GICs have certain advantages:-

1) They are still the only restorative materials that adhere to both enamel and dentine directly without the need for an intervening material (Aboush & Jenkins, 1986, 1987). Thus mechanical retention may be minimized.


3) The modern cements are relatively translucent so they can be more closely matched to enamel and dentine.

4) They are dimensionally more stable than composite and easier to use (Wilson & McLean 1988). This leads to improved sealing and reduced microleakage (Swift 1988 & McLean 1991).

However, GICs still have certain limitations:-

1) They have relatively low strength particularly in the first 24 hours after mixing and are therefore considered to be inadequate for use in high stress bearing areas (Marker et al 1985 & Walls et al 1987).

2) They show low wear resistance (Forss et al 1991 & De Gee et al 1996).

4) The immature cements are sensitive to moisture when compared with amalgam and composite (McLean & Wilson 1977c & Causton 1981).

5) The setting reaction of the cements may be inhibited by desiccation if it is placed in patients who breathe through the mouth (Anstice 1994).

6) Desiccation at a later stage will also affect the cement leading to cracking and crazing (McLean & Wilson 1988, Smales et al 1990 & Lidums et al 1993).

The use of conventional glass ionomers in stress-bearing areas in posterior teeth has been restricted by their limited strength and relatively low wear resistance. Attempts have been made to address this and have moved in two distinct directions. The first involves modifying the glass ionomer cements by the inclusion of metal particles in an attempt to improve the strength, fracture toughness, resistance to wear and to provide radiopacity. The second involves the variations in glass filler size and filler loading.

Two methods of metal inclusion have been employed, the first involves mixing with spherical or lathe-cut silver amalgam alloy powder with the glass ionomer cement powder. These are known as the admix cement (Simmons 1983). The addition of metal or alloy powder to glass ionomer cement has been shown to increase strength (Kerby & Bleiholder 1991 & Williams et al 1992a) and decrease microleakage between the cement and tooth structure (Weed et al 1986 & Chung 1993).

In the second method, silver is sintered onto the glass particles. These materials have been called cermet cements (McLean & Gasser 1985).

McLean & Gasser (1985) recommended the use of the cermet cement stating that the use of a sintered glass composition instead of silver alloy powder improved the wear resistance of the resulting cement.
This study compares the long term properties of a metal reinforced glass ionomer cement with those of its unreinforced counterpart using a disperse phase alloy amalgam as a comparator for strength and wear tests. It also evaluates the performance in a clinical trial of the metal reinforced material in comparison with amalgam.
Chapter (2)

Literature Review
2 Literature Review:

2.1 Dental amalgam:

Dental amalgam is defined as an alloy of mercury with silver, tin, copper and sometimes with zinc, palladium and indium (Craig 1989).

Amalgam is formed from two components, a powder and a liquid. The powder is an alloy in the form of fine particles, the liquid is mercury (Hg). Trituration of the alloy powder with mercury produces a plastic mass which sets by the formation of new inter-metallic compounds.

2.1.1 Types of amalgam:

There are two types of dental amalgams, conventional and high copper amalgam. Conventional amalgam is prepared from a silver-tin alloy containing small amounts of copper and zinc. The alloy should contain a minimum of 65% silver and a maximum of 29% tin with approximately 3% copper and less than 1% zinc. It is sometimes pre-amalgamated with a small proportion of mercury (Marshall 1996). The main constituent of the alloy is the gamma (γ) phase, (Ag₃Sn). This phase reacts readily with mercury to form the amalgam. Copper is present to increase the strength and hardness of the amalgam.

The size and shape of the alloy particles used are critical to the handling characteristic and the final properties of the restoration (Marshall et al 1976). Alloy powder is available as either lathe-cut or spherical particles. The lathe-cut particles are produced by machining a solid ingot of the alloy. The powder is then available as coarse,
medium or fine grains. Spherical particles are produced by spraying melted alloy into an inert atmosphere. The alloy then solidifies as small spherical pellets of various size. These are generally substantially smaller than lathe-cut alloy particles.

In the early 1960s, attempts were made to increase the strength of dental amalgam by increasing the copper content of the alloy (Youdelis & Innes in 1962). The idea was that the copper would act as a dispersed strengthening agent. High copper amalgam is prepared from either the reaction of mercury with a mixture of silver-tin and silver-copper alloys (admixed alloys) or from the reaction of mercury with a ternary spherical silver-copper-tin alloy (single composition alloys). The amount of copper in an admix amalgam is 13-20% and in a single composition amalgam is between 12-30% (Asgar 1974, Marshall et al 1976 & Eley 1997a). In the high copper amalgam the amount of silver mercury ($\gamma_1$) and tin mercury ($\gamma_2$) phases are reduced, this improves the strength and reduces the creep and corrosion. A number of examples of high copper amalgam such as Dispersalloy have been introduced by manufacturers.

2.1.2 Setting reaction:

The setting of an amalgam can be summarised as follows:-

1- Partial dissolution.

2- Precipitation of insoluble phases.

The setting reaction between the alloy and mercury is initiated on mixing the two components. The surfaces of the alloy particles dissolve into the mercury until the solution becomes saturated with respect to certain species (phases). These species are an
intermetallic compound of silver and mercury $\text{Ag}_2\text{Hg}_3$ ($\gamma_1$ phase), and a solid solution of mercury in tin which approximates to the composition $\text{Sn}_{7.8}\text{Hg}$ ($\gamma_2$ phase). The set material consist of the cores of the original alloy particles embedded in a matrix of $\gamma_1$ and $\gamma_2$ phases. Since the matrix is the weakest and softest constituent of set amalgam, the ideal structure would feature a minimum of matrix and a maximum of undissolved alloy particles (Ray 1992b).

The reaction of the conventional amalgam may be described as follows:

$$\text{Ag}_3\text{Sn} + \text{Hg} \rightarrow \text{Ag}_3\text{Sn} + \text{Ag}_2\text{Hg}_3 + \text{Sn}_{7.8}\text{Hg}$$

(\text{\textit{$\gamma$ phase}) (mercury) (\textit{$\gamma$ phase}) (\gamma_1 phase) (\gamma_2 phase) \ (1)}

powder Liquid Unreacted alloy Amalgam matrix

The reaction of admixed alloys may generalised as follows:

$$\text{Ag}_3\text{Sn} + \text{Ag-Cu} + \text{Hg} \rightarrow \text{Ag}_2\text{Hg}_3 + \text{Cu}_5\text{Sn}_5 + \text{Ag}_3\text{Sn}$$

(\text{\textit{$\gamma$ phase}) (eutectic) (mercury) (\gamma_1 phase) (\beta phase) (\gamma phase) \ (2)}

powder silver-copper liquid amalgam matrix unreacted alloy

In fact there are two reactions. The first reaction is the same as that in conventional alloys equation (1), but this is followed by a second reaction which may be described as follows:

$$\text{Sn}_{7.8}\text{Hg} + \text{Ag-Cu} \rightarrow \text{Cu}_5\text{Sn}_5 + \text{Ag}_2\text{Hg}_3$$

(\gamma_2 phase) (eutectic) (\beta phase) (\gamma_1 phase) \ (3)

The $\gamma_2$ phase is not present in many types of high copper amalgams (Marshall et al 1976 27
The single composition alloys reaction may be described as follows:

$$\text{Ag-Sn-Cu} + \text{Hg} \rightarrow \text{Ag}_2\text{Hg}_3 + \text{Cu}_6\text{Sn}_5 + \text{Ag-Sn-Cu}$$

(Ternary) (mercury) ($\gamma_1$ phase) ($\beta$ phase) (Ternary) (4)

Powder liquid Amalgam matrix Unreacted alloy

2.1.3 Properties of dental amalgam:

1- Amalgam has a high early compressive strength of more than 250 MPa at one hour (Craig 1989 & Ray 1992b).

2- Although shrinkage associated with the dissolution of the alloy particles in the mercury may occur initially, expansion occurs after 15 to 20 min. The result of these two competitive reactions is an overall shrinkage of about 10-12 $\mu$m/cm. The material is dimensionally stable in the long-term (from 6-8 hours post placement) (Ray 1992b).

3- The amalgam seals itself against leakage within a few months through corrosion process while most other materials rely on bonding or cementing procedure (Newman 1992).

4- The high copper amalgam shows less creep when compared with conventional amalgam (Mahler & Van Eysden 1969 & Mahler 1997).

2.1.4 Historical development of dental amalgam:

G. V. Black (1896) identified the alloy composition (silver and tin) which gave the best restorative grade amalgam. He described this alloy as a "balanced" composition alloy. Later, a small amount of copper was added to the alloy. This addition served to increase strength and decrease flow (Skinner 1946). The American Dental Association
(ADA) Specification No. 1 for alloy for conventional amalgam sets a maximum limit of 6% for copper content. Studies have shown that the (γ₂) phase is the weakest phase in the set conventional amalgam (Asgar & Sutfin 1965). It is the principal phase to undergo corrosive breakdown (Schoonover & Souder 1941, Wagner 1962, Jorgensen & Saito 1970 & Holland & Asgar 1974), and is responsible for creep (Mahler & Adey 1979 & Mahler et al 1982).

In 1930, the Charity Hospital in Berlin, appointed a committee to investigate amalgam toxicity. The committee declared that there was no reason to condemn the silver-tin amalgam, which was in clinical use and they also stated that the amalgam played a useful role in dentistry (Hardt 1930). It is reported that Stock (1941) stated in a lecture that the rare cases of mercury poisoning should in no way affect the use of silver amalgam in dental practices.

Youdelis & Innes (1962) proposed a compositional change in dental amalgam alloys. They experimented with the addition of ceramic powders and then with the addition of another metallic alloy to a traditional amalgam composition. Their objective was to increase the strength of the amalgam by dispersing a harder phase throughout the structure. The final selection was the addition of a Ag-Cu eutectic dispersed phase, which had the ability to form a bond through partial amalgamation with the amalgam matrix phase. This became known as a high-copper amalgam alloy which contains over 16% copper and therefore does not conform to the ADA Specification limit of 6% (Mahler 1997). The addition of the Ag-Cu eutectic changed the composition of the final amalgam alloy by eliminating the tin-mercury phase (γ₂).
Clinical studies demonstrated clearly the superiority of disperse phase alloy (Dispersalloy), a high copper amalgam, in terms of low creep and less corrosion, over $\gamma_2$-containing amalgams (conventional alloys) (Mahler 1969, Duperon et al 1970, Binon et al 1973 & Jacobsen 1984a).

Zinc was also added to amalgam alloys and was shown to increase the corrosion resistance of $\gamma_2$-containing amalgams (Jorgensen & Esbensen 1973). In clinical studies zinc containing alloys have been found to exhibit better marginal integrity and less tarnish than the zinc-free alloys (Wilson & Rye 1963, Watson et al 1973 & Mahler et al 1980). In later clinical study zinc-free Dispersalloy demonstrated significantly poorer marginal integrity than the Dispersalloy containing zinc (Osborne & Berry 1992).

2.1.5 Advantages of high copper amalgam over conventional amalgam:

1) High compressive strength and hardness (Youdelis & Innes 1962 & Mahler 1997).

2) Superior marginal integrity due to the elimination of ($\gamma_2$) phase (Mahler et al 1980).

3) Low creep and improved corrosion resistance (Sarkar & Greener 1972).


2.1.6 Limitations of dental amalgam:

The use of dental amalgams has been the subject of considerable discussion since the introduction of possible alternatives, composite resin or glass ionomer cement. The limitations of amalgam include poor aesthetic, mercury toxicity, galvanic effects, lack of adhesion and microleakage and corrosion.
1) Poor aesthetics:

Amalgam restorations are not visually attractive because of their metallic appearance. The polished finish of conventional alloy is lost with time, due to tarnish. Although amalgam is considered to be durable in posterior restorations, there is an increasing demand from patients for more aesthetic restorations.

2) Mercury toxicity:

Dental personnel using amalgam can be exposed to mercury either in the form of vapour or as particulate amalgam dust. This can be the result of poor mercury hygiene, by incorrect storage of mercury or waste amalgam (Iyer et al 1976, Mantyla & Wright 1976 & Eley 1997b) or spillage of mercury or waste amalgam used in dental procedures (Iyer et al 1976, Mantyla & Wright 1976 & Merfield et al 1976).

Mercury exposure can also occur during dental procedures. The total amount of mercury released from all these procedures has been quantified (Engle et al 1992) as follows:

1- Preparation of amalgam, (Trituration) 1-2 μg
2- Placement of amalgam restorations. 6-8 μg
3- Remove of amalgam restoration under water spray & high volume suction 15-20 μg
4- polishing of amalgam resonations, Dry polishing 44 μg
   Wet polishing 2-4 μg
5- Additional evacuation for 1 minute to remove residual amalgam dust 1.5-2 μg

It has been shown that a number of workers exposed to mercury have developed skin hypersensitivity to mercury (Langworth et al 1993).
Another concern is that mercury is released from amalgam restorations and absorbed into the body in such quantities as to be responsible for a wide range of health problems (Eley & Cox 1993 & Tyas 1994). Cases of mercury allergy have been reported (approximately 50 since 1906), but more commonly, local soft tissue lesions adjacent to amalgam restorations which resolve on removal of the amalgam have been reported (Munksgaard 1992 & Tyas 1994). Rowland et al (1994) examined the effect of occupational exposure to mercury on the fertility of female dental assistants. Their conclusion was that women with high occupational exposure to mercury were less fertile than unexposed controls.

There is some ingestion of mercury on placing a freshly mixed amalgam. The urine mercury levels are low but these levels are higher in patients with amalgam fillings than those without (Olstad et al 1987). However, there is some variation in blood mercury levels but there are no consistent difference in levels between subjects with and without amalgam fillings (Abraham et al 1984 & Snapp et al 1989). Olstad et al (1990) in later study has also found no difference in urine mercury levels in patients before and after amalgam placement. All these findings have increased the public concern. This has in turn led to a demand for a better alternative material.

Recently, Eley (1997) has published a series of articles, on the future of dental amalgam, and concluded that the only adverse effect documented is the hypersensitivity to mercury in a very tiny proportion of the population. It is, however, recommended that in continuing to use amalgam, dentists must use strict mercury hygiene procedures to avoid risk to their staff and contamination of the environment. It seems that mercury
contamination of the environment is likely to be the main reason for any future
government action against the continued clinical use of dental amalgam (Eley 1997c).

3) Galvanic effects:

If two metallic restorations containing metals of differing electronegativities are
placed in close proximity to one another in an electrically conducting medium (saliva) or
gold crown opposite to amalgam filling, it is possible that a galvanic cell will be set up.
The current generated can cause patient discomfort or leave a strong metallic taste in the
mouth. It can also accelerate corrosion of the restoration.

4) Lack of adhesion and microleakage:

The retentive cavity designs needed for dental amalgam restorations often requires
a large amount of sound enamel and dentine to be removed. This principle is
questionable, because amalgam can never be a true substitute for healthy tooth structures.
New concepts in cavity preparation aimed at minimising the loss of healthy tooth have
been developed, but the use of amalgam can never be as conservative as the approach of
using adhesive restorative materials (Van Noort 1994a).
The lack of adhesion also encourages microleakage, which may contribute to restoration
failure. Recurrent caries is one of the major problems associated with amalgam
restoration. Mjör (1985) reported a study investigating (over a two weeks period) the
reason for replacement of amalgam restorations in general dental practices, placed by 28
clinicians. In that fortnight, 72% of the restorations that were replaced were diagnosed
as having recurrent caries. The recurrent caries had developed underneath the restorations
in less than 10 years.
5) Corrosion:

Corrosion of amalgam is the process by which metals including mercury can be released from amalgam restorations. It is thus relevant to mercury release from amalgam restoration during function (Eley 1997a). It is well recognised that amalgam corrodes in the oral environment. The $\gamma_2$ phase is considerably more electronegative than the $\gamma$ and $\gamma_1$ phases. This means that in the presence of an electrolytic solution, the $\gamma_2$ phase will act as the anode of the oxidation cell and will gradually dissolve. This process will severely weaken the amalgam structure and is often cited as a cause of marginal breakdown. However, corrosion products may be useful in sealing defective margins and in anchoring the restoration to the tooth.
2.2 Composite resin cement:

Composite resin is a tooth coloured translucent restorative material.

2.2.1 Composition:

The constituents of a composite resin cement include high and low molecular weight monomers, inorganic fillers, a silane coupling agent and polymerisation initiators. The resin forms the matrix of the composite material binding the individual filler particles together via the coupling agent.

1) The resin matrix (monomers):

The matrix is the chemically active component of the composite. It is initially a monomer, but is converted into a rigid polymer by a radical addition polymerisation. The most commonly used monomer is the reaction product of bisphenol-A and glycidyl methacrylate, known as Bis-GMA, because its large molecular size ensures a low polymerisation shrinkage. However, Bis-GMA is highly viscous, which makes it less suitable for dental composite as the addition of inorganic filler particles results in very tough pastes, whose handling properties are not acceptable in dental clinic. Therefore, Bis-GMA is diluted with a lower molecular weight, lower viscosity methacrylate. The most common diluting methacrylates are ethylene glycol dimethacrylate (EGDMA) and triethylene glycol dimethacrylate (TEGDMA). A possible formulation would be 75% Bis-GMA and 25% viscosity controllers (Watts 1992). Urethane dimethacrylate (UDMA) resin has been used as alternative to Bis-GMA in a number of composites, because UDMA is less viscous (Pearson 1990).

The resin matrix also contains an inhibitor (hydroquinone) and activator/initiator systems.
for initiating the cure, either by chemical or light activation.

2) The filler:

The filler used in the early composites was quartz. High filler loadings up to about 70% by weight, with particle sizes up to a maximum of 70 μm were used. More recently most composites employ one or more of a variety of glass fillers including colloidal silica and silicate glasses containing barium or strontium (Pearson 1990). The inclusion of barium or strontium provides radiopacity for the cement, and this aids the detection of recurrent caries. The silicate glasses are considerably softer than quartz which improves the surface finish of the composite.

The particle size of the filler is important as it will influence the amount of filler that can be added to the resin, without the necessary handling characteristics being lost. It has also an effect on the final surface finish of the restoration, in that the smaller the filler particle size the smoother the composite will be.

The shape, loading, size and distribution of filler in composites determine the level of wear of composite resins (Hirt et al 1984) and properties such as compressive strength and stiffness (Van Noort 1994).

3) Silane coupling agents:

Silane coupling agents are used in composite materials to provide a strong chemical bond between the oxide groups on the glass surface (filler) and the polymer molecules of the resin matrix (Van Noort 1994). The potential advantages of such a matrix-particle bond are increased rigidity and strength due to the reinforcing action of the filler (Bowen & Rodriguez 1962). Silane coupling agents improve fracture toughness,
resin wetting of the filler and protect the filler particles against stress corrosion by water (Soderholm 1981). They also improve optical properties because they reduce the scattering of light at the resin-filler interface (Soderholm 1983).

Two commonly used silane coupling agents are γ-aminopropyltriethoxysilane and γ-mercaptopropyltrimethoxysilane.

2.2.2 Composite classification:

Composite resin materials may be classified according to the type of filler included in their formulation, as follows, (Ray 1992a & Van Noort 1994b):

1) Macrofilled composites:

These materials have high inorganic filler loading, up to about 86% by weight. These contain glass filler particle size of 10-20 μm and a largest particle size of 40 μm. Macrofilled composites have reasonable mechanical strength and wear resistance. However, these composites had the disadvantages that the surface finish was very poor, with a dull appearance and difficult to polish. These materials showed two different patterns of wear, an interfacial defects and general loss of material over the occlusal surface (Leinfelder et al 1989). The average value of wear obtained with ACTA wear machine was 40 μm per 200,000 rev (De Gee & Pallav 1994).

2) Microfilled composite:

The filler in the microfilled materials is colloidal silica and the mean particle size 0.04 μm. The filler is mixed with the monomer and the mixture is then polymerised in order to form prepolymerised filler blocks. These blocks, ground down to particulate
form, are then mixed with monomer to form the cement paste. The total inorganic filler loading is reduced to about 50% by weight. This results in decreased mechanical strength and wear resistance, but the softer filler ensures that the surface can be easily polished. These materials are suited to unloading bearing areas where good aesthetic are important. These materials showed different patterns of wear, a marginal fracture at the restoration tooth interface and local wear in the occlusal contact area (Leinfelder et al 1989). The average value of wear obtained with ACTA wear machine was 49 μm per 200,000 rev (De Gee & Pallav 1994).

3) Hybrid or blended composite:

Hybrid composites were developed in an attempt to combine the polishability and superior aesthetics of the microfilled cements with the improved mechanical properties of the macrofilled materials. The filler is a combination of that used in both materials. The total inorganic filler loading may approach 80% by weight. The large variation in filler particle sizes is considered to yield a surface which is less susceptible to wear than the macrofilled material (Ray 1992a).

The hybrid composites showed different patterns of wear, a marginal fracture at the restoration tooth interface, localized wear in the occlusal contact area and moderate loss of material over the occlusal surface (Leinfelder et al 1989). The average value of wear obtained with ACTA wear machine was 25 μm per 200,000 rev (De Gee & Pallav 1994).

2.2.3 Composite setting reaction:

The setting reaction of composite is a free-radical polymerisation of the monomer. In the set cement the filler particles embedded in the matrix are bonded to the matrix via
silane coupling agents. The polymerisation may be initiated either by mixing using a
chemical-cured initiator system or by exposure to an intense light of an appropriate
wavelength using a visible light curing initiator system (VLC). The self cure initiator
system is usually benzoyl peroxide coupled with a tertiary amine. The visible light cure
systems employ an \( \alpha \) diketone (benzil or camphorquinone) as the photosensitisable, free-
radical initiator with a tertiary amine as a reducing agent. The initiating light is blue
operating at a wavelength between 460-470 nm (Taira et al 1988).

2.2.4 Adhesion of composite resins:

Adhesion is generally defined as, the state in which two surfaces are held together
by interfacial forces which may consist of chemical forces or interlocking forces
(mechanical) or a combination of both (Retief & Denys 1989).

Dental composites are hydrophobic materials with no capability of bonding to
tooth structure. The mechanism of retention of the restoration within the cavity must be
mechanical in origin or alternatively the tooth must be treated with an appropriate
solution to allow chemical interaction (Erickson & Glasspoole 1994)
Adhesion must be achieved to both enamel and dentine. Enamel is an acellular almost
totally inorganic crystalline structure whereas dentine has both inorganic and organic
material together with water as constituent.

1) Mechanical adhesion to enamel:

The acid etch technique concept was first introduced by Buonocore (1955). This
technique combined with the development of composite resins (Bowen 1963), gave rise
to the acid etch technique for composite resins.

Silverstone (1974) and Silverstone et al (1975) described three types of patterns obtained by etching enamel surface with orthophosphoric acid: - Type I, preferential dissolution of the prism cores occurs which results in a typical "honeycomb" appearance. Type II, preferential dissolution of the prism peripheries occurs, resulting in a "cobblestone" appearance. Both types I and II patterns are often obtained in adjacent areas on enamel in which the prisms extend to the enamel surface. In the type III pattern, surface loss occurs without exposure of the underlying prism.

After etching, the curing resin flows by capillary action into microporosities (channels) in the enamel. This produces resin tags up to 50 micrometres which will bond by micromechanical means to the etched enamel after polymerisation (Prevost et al 1984 & Retief 1987). Etching the enamel surfaces with phosphoric acid (30% to 50%) for either 15, 30, or 60 seconds have been recommended (Retief & Denys 1989, Garcia-Godoy & Gwinett 1991).

The effect of what is resin impregnation of the enamel is that there is substantially reduced microleakage at the tooth/restorative interface provided that sufficient thickness of enamel is present (Retief et al 1982).

2) Adhesion to dentine:

Dentine consists of approximately 70% by weight of inorganic, in the form of hydroxy-apatite, 12% water and 18% organic, mainly collagen, arranged as intertubular and peritubular dentine around tubules (Griffiths & Watson 1995).

Acid etching of dentine removes the smear layer (a layer of debris remaining on instrumented tooth surfaces) and opens dentinal tubules thus allowing mechanical
interlocking between the restoration and the dentine to be achieved, but this retention was originally thought to be insufficient and should be supplemented by some form of chemical interaction (Asmussen & Munksgaard 1988). Intensive investigations have therefore been directed towards the development of the dentine bonding agents (DBAs). These are di- or multifunctional organic molecules, in which one active group bonds to the dentine and the other, often a methacrylate, copolymerizes with the restorative material (Nakabiyashi et al 1982). The bonding agent may react with either the inorganic or the organic component of the dentine using either ionic or covalent bonds respectively.

Inorganic bonding can be initially achieved using phosphonate groups as the functional group of the adhesive. The bonding with the inorganic phase is by electrostatic attraction between the negatively charge phosphate group and the positively charged calcium on the surface of the dentine and also chelation of the calcium ion by the bonding agent via oxygen atoms. The bond strength to dentine can be increased by treating the surface with a solution of mordant ions (calcium or ferric ions) (Causton 1984).

The organic groups available to interact with a bonding agent are the amino acids, hydroxyl, amino and amido groups. The pretreatments that have been found to be effective in bonding to the organic phase of dentine (collagen) include acid chlorides, acid anhydrides and isocyanates. However, these are all moisture sensitive and as such may be a contributory rather than fundamental mechanism in the adhesion process (Asmussen & Munksgaard 1988).

DBAs reacting with the inorganic phase have shown in vitro bond strengths
approaching those to etched enamel (Retief et al 1988). They consist of three components: a conditioner, a dentine primer and adhesive resin. The conditioner removes the smear layer, the primer infiltrates the surface and helps to link the resin to the dentine surface and the adhesive resin applied on tops is a light cured resin containing Bis-GMA and hydroxyethyl methacrylate (HEMA).

The exception to this is GLUMA which contains a glutaraldehyde and HEMA bonding system. This has been used in bonding to the organic phase. The dentine conditioner is a buffer solution of ethylene diamine tetra-acetic acid (EDTA) which is designed to remove the smear layer. The dentine primer contains a mixture of glutaraldehyde which fixes collagen and HEMA which primes the tooth surface for subsequent adhesion by the resin. The bonding resin consists of visible light cured mixture of Bis-GMA and TEGDMA (Kanca 1989). The bonding reaction is thought to be initiated by attack of the aldehydes on the amino groups of the collagen. A complex is formed by this process which can react with the hydroxy group of the methacrylate monomer, linking the methacrylate group to dentine.

New findings have suggested that dentine etching may play a role in mechanical bonding, which was principally micro-mechanical in nature (Erickson 1989). The adhesive is reported to flow into etched dentine tubules and form a mechanical bond (Griffiths & Watson 1995). The modern systems provide a means of partially demineralisation the peritubular dentine permitting low viscosity primers to impregnate the outer few micrometres of dentine (Van Meerbeek et al 1993, Perdigão 1995 & Griffiths & Watson 1995).
2.2.5 Biocompatibility:

The acid etchant appears to open the dentinal tubules, but is not apparently harmful in itself. However, acid etching permits a greater penetration of the low-viscosity bonding agent which contains potentially irritating low molecular weight monomers. Composite is reported to show mild to severe pulpal irritation where the remaining dentine is less than 1 mm thick (Craig 1989). Consequently pulpal protection should be used where tubules are most likely to communicate directly with the pulp.

It has been reported that the bisphenol-A and bisphenol-A dimethacrylate components of one commercial resin-based composites and sealants used in dentistry are oestrogenic and may represent an additional sources of human exposure to xenoestrogens (Olea et al 1996). However, a recent study investigated the potential oestrogenic effects of bisphenol-A in seven sealants, and showed that no estrogenic effects can be detected from any of these sealants (Hamid & Hume 1997).

2.2.6 Limitations of composite:

1) Composite polymerisation shrinkage:

Polymerisation shrinkage has long been recognised as a serious drawback with composites. In a sense, the whole field of adhesive restorative dentistry grew from this limitation. The polymerisation shrinkage of a composite is dependent upon the type of resin employed and the amount of resin present in its unpolymerised form. The polymerisation shrinkage of the composite leads to poor marginal adaptation and increases the possibility of recurrent caries.

Another potential problem is that the shrinkage will cause the cusps of the tooth to be pulled inwards so that they become highly stressed and enamel fracture is likely (Pearson
& Hegarty 1987 & 1989). This effect may lead to postoperative sensitivity which is associated with leakage.

There have been several attempts to solve the shrinkage problem. The most logical and effective one is the use of low-shrinkage monomers such as Tetrahydrofurfuryl-methacrylate (THFMA) (Patel et al 1987) or Spiro-orthocarbonates (Eick et al 1993). Development are currently taking place in this area.

The unpolymerised methacrylate groups which exist in the set cement as residual monomers can act as plasticiser reducing the mechanical properties of the cement (Watts 1992). *In vitro* studies have shown the release of unpolymerised monomers present in dental composites over at least the first 24 hours after setting (Ferracane & Condon 1990). Enzymatic hydrolysis of methacrylates together with mechanical forces, also contribute to the gradual breakdown of the cured composites (Freund & Munksgaard 1990 & Ripa 1993). This breakdown may also lead to pulp sensitivity.

2) Composite wear:

Wear is a major factor that has restricted the use of dental composite resins in class I and II restorations, where they are subjected to greater occlusal loads and abrasive action during mastication (Phillips et al 1973 & Hirt et al 1984). Composite wear has two phases. First, microabrasion of the polymer occurs under stress and under the action of abrasive food. This causes the filler particles to be exposed and to be increasingly subjected to mechanical stress. The second phase is the dislodgement of the filler particles (Ehrnföld et al 1980 & McKinney & Wu 1982). Hirt et al (1984) reported that the level of wear of composite resins is determined not only by the filler type and the filler matrix interface but also by the quality of the resin.
Draughn and Harrison (1988) concluded that wear resistance of filled composite resin was dependent on the size, proportion by volume and hardness of the filler particles. The degree of cure, filler level and silanisation level also affect the wear resistance of composites (Condon & Ferracane 1997).

O’Brien & Yee (1980) reported that wear of composite resin could be the result of the following mechanisms:

1) Wear of the resin matrix.

2) Adhesive failure: loss of filler by failure of its bond to the matrix.

3) Shear of filler particle: loss of filler through shearing of exposed particles.

4) Cohesive failure: loss of filler through cracking and failure of the matrix.

5) Exposure of the entrapped air bubbles in the resin.

3) Composite strength:

The compressive strength of posterior composite resin is between 250-320 MPa (Craig 1989). This strength is lower than that of amalgam which ranges between 380-500 MPa (Malhotra & Asgar 1978). The diametral tensile strength of composite resin is in the range between 35-40 MPa (Craig 1989). This is relatively lower than that of amalgam which ranges between 49-58 MPa (Malhotra & Asgar 1978). Transverse strength more frequently referred to as flexural strength shows composite having a value of 139 MPa with lathe-cut alloy 124 MPa.

4) Water sorption:

Composites are moisture sensitive, so water contamination during placement may affect the material properties. Water sorption occurs mainly as a direct absorption by the
resin. Excessive levels of water sorption have detrimental effects on colour stability and
wear resistance. Incorporation of water will also lead to plasticisation of the resin and
consequential reduction in mechanical properties.

5) Radiopacity:

The detection of caries under a non-radiopaque composite is virtually impossible
and would allow the caries process to continue undetected for far too long. Some
composites have a radiopacity lower than that of dentine which is also inadequate.
However, it is not clear what the optimum radiopacity for a composite is, since excessive
radiopacity could potentially mask out caries around the restoration. The British and
International Standards accept a radiopacity equivalent to 2 mm of aluminium (BS 5199:

6) Recurrent caries:

It has been reported that there is a high level of occurrence of secondary caries
beneath composite materials (Forsten et al 1982). This is possibly due to plaque
accumulation. Mjör (1985) found a greater accumulation of plaque on composite
materials, when compared to that on amalgam, located along the incisal or occlusal part
of the restoration. Mjör (1996) reported also that a nearly 1:5 relationship was found
between composite restoration placed to treat primary caries and those placed to replace
failed restorations due to secondary caries.
7) **Mismatch of thermal properties:**

The coefficient of thermal expansion per °C of composite is different from that of teeth, [tooth crown is $11.4 \times 10^{-6}$ and composite is $30 \times 10^{-6}$] (Craig 1989). This, in combination with the gap resulting from polymerisation shrinkage, can increase the potential for percolation of oral fluid along the interface. When a tooth containing a composite is chilled, the restoration tends to contract more than the surrounding tooth structure, enlarging the space between the two. This allows greater fluid percolation. After the stimulus, the temperature of the oral cavity returns to 37°C, the restoration expands and the fluid is forced out of the space between the tooth and the restoration. These cyclic changes can lead to material fatigue and early bond failure (Craig 1989).

Composite is considered to be an alternative aesthetic restoration. However, the drawbacks to its use include lack of abrasion resistance and dimensional instability which stresses the bond sealing the cavity against recurrent caries. These, with other limitations discussed previously, encourage research for a better alternative.
2.3 Glass ionomer cement:

2.3.1 Background:

The 1983 British Standard (BS 4492) defined a dental cement as either;
- a non-metallic dental material used for luting or filling for permanent or temporary restorative purposes, and made by mixing components into a plastic mass which sets.
or
- the product of an acid-base reaction, formed by the interaction between a base (in powder form) and an acidic liquid. A salt hydrogel is formed, which causes the cement to set and harden.

The glass ionomer cement (GIC) comes under the second definition (Wilson 1978 & Wilson & Nicholson 1993). The GIC is formed by the reaction of an acid degradable aluminosilicate glass powder (the base) with a concentrated solution of polymeric acid. The difference between the GIC and a composite material is the role of the filler in the setting reaction. GIC has glass particles which are fundamental to the reaction mechanism whereas the filler particles of a composite resin do not undergo any chemical change during the reaction (Watts 1992).

2.3.2 Glass ionomer cement original formulation:

GICs were developed at the Laboratory of the Government Chemist by Wilson and Kent, during the late 1960s and early 1970s.

1) The Glass:

The three essential constituents of the glass are silica (SiO$_2$), alumina (Al$_2$O$_3$) and calcium fluoride (CaF$_2$).
The original composition of the silicate glass powders used in the GICs was based on the formulation:

$$\text{SiO}_2\cdot\text{Al}_2\text{O}_3\cdot\text{CaF}_2\cdot\text{AlPO}_4\cdot\text{Na}_3\text{AlF}_6$$

Calcium fluoroaluminosilicate glass is the most common glass used in these cements. It is prepared by fusing quartz, alumina, aluminium trifluoride, calcium fluoride and aluminium phosphate at a temperature between 1100°C and 1500°C. The glass is shock quenched then ground to a fine powder. During firing of the glass, aluminium can enter the silica network replacing silicon in the structure. This causes the network to becomes basic and hence susceptible to attack by hydrogen ions from the acid. The alumina/silica ratio of the glass is required to be 1:2 or more by weight for this to occur (Kent et al 1979 & Wilson et al 1980).

Fluoride is an essential constituent of the glass. It reduce the temperature of fusion of the glass, improves the working and setting times of the cement, increases the strength of the set cement and enhances the translucency of the cement. Its steady release contributes to the prevention of tooth decay (Wilson & McLean 1988).

Glasses high in silica (greater that 40%) are transparent, whereas glasses high in calcium fluoride or alumina are opaque. Current commercial products contain fluorite or corundum to enhance opacity and/or a variety of components to achieve radiopacity including strontium, barium & lanthanum salts, zinc oxide (McLean & Gasser 1985). However, achieving the radiopacity may affect the cement performance.
2) The Acid:

The liquid component of GIC was originally aqueous polyacrylic acid (PAA) (Wilson & Kent 1971). A wide range of polyacids based on the homo or copolymers of a number of alkenoic acids with acrylic acid are now used including itaconic and maleic acids (Crisp & Wilson 1974a, 1977, Crisp et al 1980, Wilson & McLean 1988 & Schmidt et al 1981). The molecular weight of the polyacid lies between 10,000 and 55,000 depending upon the formulation selected.

The problem with the original polyacrylic acid solution was gelation on storage. The segments of different chains of the polymer solution approached each other and formed hydrogen bonds between acid groups on different chains (Crisp et al 1975a, McLean & Wilson 1977b). The formation of hydrogen bonds had the effect of slowly cross-linking the PAA. The mobility of the chains in solution decreased as the density of cross linking increased and with time the solution gelled. The copolymer molecules are less regular than those of the homopolymer and are less liable to gel (Crisp & Wilson 1977b). This is because the irregularity of the copolymer impedes the molecule-molecule interaction required for the formation of the hydrogen bonds.

Schmidt et al (1981) investigated an acrylic/maleic copolymer in the ratio 1:1 and found that the copolymer’s adhesion was comparable to that obtained for polyacrylic acid. However, Aboush & Jenkins (1986) found that the bond strength to enamel and dentine of a cement based on polyacrylic acid was higher than that of a cement based on a copolymer with maleic acid. It is possible that the irregular structure of the copolymer limits interaction with the tooth surface.
Increasing the molecular weight of the polyacid is known to increase the strength of the set cement and to decrease the setting time (Prosser et al 1986). However, higher molecular weight polyacids are more difficult to get in solution. An alternative method is to use vacuum dried forms of the high molecular weight polyacid (anhydrous GIC). The liquid for cement formation is then either plain water or an aqueous solution of tartaric acid. However, this anhydrous formulation has been shown to lead to incomplete dissolution of the vacuum dried acid and subsequently to the formation of a weakened cement (Guggenberger et al 1996).

The addition of tartaric acid to the liquid has been reported to improve the handling characteristics of the cement, (extending the working time and accelerating the setting reaction) and to increase the cement strength (Crisp & Wilson 1976, Wilson et al 1976, Prosser et al 1982, 1982a & Cook 1983). Since tartaric acid is a stronger acid than polyacrylic acid (PAA), it reacts preferentially with the glass and complexes the metal ions extracted from it more readily than PAA (Crisp & Wilson 1976, Prosser et al 1982 & Hill & Wilson 1988). Thus, tartaric acid prevents the early binding of cations to the polyanion chains (Prosser et al 1982). It also controls the pH during the setting process which in turn controls the rate of dissolution of the glass (Wilson & McLean 1988). Crisp and Wilson (1976) also proposed that tartaric acid may play a structural role in the cement by forming bridges between pairs of metal ions and hence, link the polymer molecules. More significantly, the use of tartaric acid widens the range of glasses that can be used for cement formation (Nicholson et al 1988). The low viscosity of these cements on mixing allows easy flow and makes them suitable for use as luting or lining cements.
2.3.3 Setting reaction:

The setting reaction of glass ionomer cements has been described (Kent et al 1973, Crisp & Wilson 1974a, 1974b, 1976 & Crisp et al 1974, 1975) (Fig 1) as follows:-

1) Decomposition of the glass and migration of ions.
2) Gelation.
3) Hardening and slow maturation.

On addition of the liquid to the powder, hydrogen ions from the polyacrylic acid solution rapidly attack the surface of the glass particles (Wilson & Kent 1972) causing complete dissolution of a fraction of the glass surface (Wasson & Nicholson 1990) causing the release of cement forming ions, mainly calcium and aluminium into the cement paste (Prosser & Wilson 1979 & Swift 1988). In the first instance calcium ion salt bridges alone appear to be formed between carboxylate groups of the polymer, resulting in a gel matrix surrounding the intact glass particles. This gives the initial set of the material (Crisp & Wilson 1974b) which was later confirmed by Nicholson et al (1988). At this stage, both water uptake and water loss can occur.

The aluminium salts are formed later and are responsible for the final hardening of the cement (Crisp & Wilson 1974a, Crisp et al 1974 & Bowen & Marjenhoff 1992). The maturation of the GIC is relatively slow, because aluminum salts form more slowly. The rates of salt formation are different because the aluminium species are likely to be released from the glass in the form of a complex species containing a number of aluminium atoms. This complex will only gradually breakdown to release discrete aluminium ions required to react (Wasson & Nicholson 1990).
The release of orthosilicic acid, which accompanies the release of glass ions, has been observed and during the setting reaction is converted to silica gel (Crisp et al 1974), which may also contribute to the hardening process in the form of a secondary setting reaction (Wasson & Nicholson 1990, 1991 & 1993).


2.3.4 Factors affecting the setting reaction:

Early GICs suffered from poor setting characteristics, they had a limited working time and delayed hardening (Wilson 1989).

The factors which may influence the setting reaction of GICs are as follows:-
1) Glass composition:

The setting reaction is determined by the nature of the glass, which has to be sufficiently basic to be decomposed by the polymeric acid. As previously stated glass basicity is controlled by the alumina/silica ratio. Within a certain limit the ratio affects the setting reaction, the higher the alumina/silica ratio the faster the set and the shorter the working time (Kent et al 1979 & Wilson et al 1980). If the value of the ratio is too low, the glass is unreactive and cement formation does not occur. The rate of setting of the cement can be varied by acid treatment of the glass. Acid treatment of the glass involves washing the glass with mineral or organic acids. It is a process which reduces the amount of calcium available for extraction during the early stages of the reaction, and thus prolongs working time (Schmidt et al 1981).

2) Particles size of the glass powder:

The smaller the particle size, the greater the surface area resulting in faster setting materials with shortened working and setting time (Kent et al 1979). In addition, the particle size of the glass affects the resistance to erosion, aesthetics and the cement strength. The median particle size of conventional GIC (Ketac Fil) is about 5 μm (Guggenberger et al 1996). However, high abrasion resistance is reported to have been achieved by decreasing the median particle size to 2 μm (Ketac Molar). This reduction could also improve the physical properties of the cement (Guggenberger et al 1996).

3) Addition of tartaric acid:

The addition of tartaric acid as described previously has an essential influence on the setting reaction of the GIC. The most important effects of tartaric acid are the

4) Relative proportion of the constituents in the cement mix:

The constituents of the cement are glass, polyacid, tartaric acid and water. The greater the proportion of the glass and the lower the proportion of water, the faster the set and the shorter the working time (Crisp et al 1976b).

5) Temperature:

The temperature of the cement paste can be controlled by the clinician. The higher the temperature, the shorter working time and the faster the set. One of the techniques which has been employed to increase the working time of the GIC is chilling of the powder and mixing slab (Mount & Makinson 1982). This effect was more noticeable with water-base cement and elevation of the component temperature did not greatly shorten the setting time (Pearson & Atkinson 1987). However, condensation of water on the mixing slab may affect the water balance of the cement.

2.3.5 Role of water in the setting reaction:

GICs are sensitive to presence of moisture during the initial stages of the reaction, when the cement forming salts are in a soluble form. In the initial stages of set, exposure to moisture may remove the cement-forming ions (aluminium and calcium), and severe weakening of the cement will occur (McLean & Wilson 1977c).
Water plays two important roles in the GIC's setting reactions:

1) **Transportation:**

Water provides the ion transport system needed for the acid-base setting reaction to take place. A reduction in water content speeds up the rate of reaction, but a minimum concentration of water is required for the reaction to proceed.

2) **Hydration:**

Water also provides the means of hydration of the siliceous hydrogel and the metal polyacrylate salts formed during the setting reaction. The water present in the set cement is in two forms. The first form is loosely bound water which is easily removed by desiccation and the other form is tightly bound water which can not be removed in this manner (Wilson & Crisp 1975, Elliot et al 1975, Crisp et al 1976c & Wilson et al 1981). The amount of loosely bound water decreases with time whereas the proportion of tightly bound water increases. The ability of the cement to absorb or lose water decreases but is not eliminated as it ages (Saito 1978 & Hornsby 1980).

GICs are sensitive to moisture during the initial stages of the reaction. Exposure to moisture may weaken the cement and cause it to lose its translucency (Causton 1981). Equally, dehydration at this stage may also result in the loss of the water of hydration and crazing of the cement (Wilson & McLean 1988). This is very much dependent on the type of cement, maturation and manufacturer. They may not be completely resistant to water loss for two weeks following placement (Sulong & Aziz 1990), and slow setting cements may take up to six months to be fully mature (Mount 1994).

A coat of varnish or light-cured unfilled resin should be applied to the set cement to

2.3.6 Properties of glass ionomer cement:

Characterisation of the GIC shows that it combines certain favourable properties of both the dental silicate and polycarboxylate cements, which are as follows:

1) Strength:

The strength of GICs is very variable. Initially GIC has a relatively low compressive strength, but it increases with time. Three different patterns of strength change have been identified:

- an immediate increase in strength within the first 24 hours continuing to rise which then stops at a certain point (Crisp et al 1976a).

- an increase in strength which continue for several months before plateauing (Williams & Billington 1989 & 1991 and Pearson & Atkinson 1991).

The previous two patterns have been attributed to the continuous formation of polysalt complexes during an ongoing acid-base reaction (Crisp & Wilson 1974), or to the slow rate of cement maturation (Crisp et al 1976a, Simmons 1983 & Williams et al 1992a).

- an immediate increase in strength over the first 24 hours which then plateaus before declining (Smith 1968 & Nicholson & Abiden 1997). This is possibly attribute to the hydrolytic instability (Crisp et al 1976 & Smith et al 1988). Recently, Nicholson and Abiden (1997) demonstrated that it appeared to be attribute to the higher cross-link density that can be developed in these cement and makes it brittle. This explanation may apply to the acrylic/maleic acid copolymer cements.

It has been suggested that the strength of GICs may be significantly increased by the
incorporation of metal into the formulation, either by direct mixing of metal powder with the glass (Simmons 1983, Sarkar et al 1990 & Kerby & Knobloch 1992) or by fusing the glass with silver before mixing, forming a ceramic metal hybrid known as a cermet (McLean & Gasser 1985). These will be discussed in detail later in the reinforced GIC section.

2) Dimensional stability:

GICs shrink slightly on setting, this can be accentuated by loss of water. At high humidity condition the cement will absorb water which tends to cause a small expansion of the cement. Generally the expansion is sufficient to exceed any setting shrinkage (Hornsby 1980). More recent measurements suggested shrinkage of the order of 0.5%/first hour (Kanchanavasita et al 1995). The coefficient of thermal expansion for GIC is close to that of tooth structure and its thermal diffusivity is low, these combined with its ability to bond to both enamel and dentine (Wilson et al 1983 & Yap et al 1994), ensure that microleakage at the tooth/cement interface is minimal. The GIC has been reported to be dimensionally stable in the humid clinical environment (Hicks et al 1986).

3) Adhesion:

There are two mechanisms of adhesion in dentistry, chemical and Mechanical. They may be used independently or in combination. In many cases both may occur together. Chemical adhesion involves bonding at the atomic or molecular level. Mechanical adhesion is based on retention by interlocking or the penetration of one phase into the surface of the other.
GIC has the ability to bond dynamically to both dentine and enamel via both the hydroxyapatite (inorganic) and collagen (organic) components of these tissues. This union between the cement and dentine or enamel is achieved through an ionic exchange at the interface, leading to the development of an ion-enriched layer of cement which is firmly attached to the tooth (Wilson et al 1983). It is believed that the polyacrylate chains enter the molecular surface of the hydroxyapatite, displacing and replacing surface phosphate. Calcium ions have to be displaced equally with the phosphate ions to maintain electrical equilibrium and these displaced ions reinforce the underlying layer of the cement. The bond to the enamel is stronger than that to dentine which indicates that the mode of bonding is principally to the inorganic (apatite) phase (McLean 1980), although in vitro studies have shown that GICs do adhere to collagen (Beech 1973). It proposed that the initial bonds are hydrogen bonds formed by carboxyl groups in the wet paste. As the cement matures the hydrogen bonds are replaced by ionic bonds with the cations coming from either the hydroxyapatite or the cement (Wilson 1974 & Wilson & Prosser 1984). It has been suggested that after wetting of the tooth surface, molecular bridges of an ionic nature develop between negatively charged carboxylate groups in the water-suspended polymer of the cement and positively charged calcium ions in the dentine and enamel (Coury et al 1982, Powis et al 1982 & Wilson et al 1983).

The adhesion of a GIC may be enhanced by surface conditioning with 25% solution of polyacrylic or tannic acids prior to placement of the cement, and due to the dynamic nature of the GICs, adhesion tends to improved with time (Wilson & McLean 1988). Adhesion may also be enhanced by the use of fresh cut dentine (Causton & Johnson 1979). This however, has not been observed in a later study by Aboush and Jenkins (1983).
Adhesion and the potential for microleakage with the use of GICs has been discussed at length in the literature. On the one hand several studies support their use for restorations, showing that long-term adhesion to dentine and enamel can be expected with a high level of confidence (Mount 1981, Wilson & McLean 1989 & Osborne & Berry 1990). GICs provide a good marginal seal which eliminates the development of secondary caries and permits the material to be used in the restoration of cavities with no undercuts or retentive features, in cervical erosion cavities and for the sealing of pit and fissures (McLean & Wilson 1974, 1977a & 1977b & Tyas & Beech 1985). On the other hand workers have shown marginal leakage in vitro with the inference that failure in the oral cavity should be expected (Crim & Shay 1987, Barakat et al 1988, Scherer et al 1989, Cheung 1990, Mathis et al 1990 & McInnes et al 1990).

Powis et al (1982) investigated the significance of utilizing the smear layer, and found that it significantly improves bonding of GICs to dentine. However, it should be noted that in vivo the situation is complicated by fluid outflow from open dentinal tubules which will adversely affect adhesive penetration (Pashley & Depew 1986). Therefore, smear layer can eliminate the fluid outflow (0.6 μl/sq.mm/day) as reported by Paterson and Watts (1990).

Surface conditioning:

A study by Aboush and Jenkins (1986) using freshly prepared dentine has cast doubts about the clinical value of surface conditioning. However, currently, the procedure for surface conditioning recommended by most manufacturers is to apply a solution of 25% polyacrylic acid for 10 seconds or 10% for 20 seconds, then wash off and gently
dry the tooth with warm air (Mount 1994 & Yap et al 1994). The tooth surface must not be dehydrated (Mount & Makinson 1982). The polyacrylic acid has been shown to partially remove the smear layer and increase the cements bond to tooth structure (Barakat & Powers 1986, Hinoura et al 1986 & Mount 1994).

4) Fluoride Release:


Fluoride is an essential constituent of the GIC and is released over a long period of time. The rate of release is constant by the second year (Forsten 1990 & 1991), continues for at least seven years (Mount 1996) but probably goes on for the life of the restoration. Tay and Braden (1988) postulated that the fluoride release involved two processes. Initially fluoride ions are released from the surface as a washout process and then from the bulk of material by diffusion. The surface degradation produces an initial burst of fluoride while the bulk diffusion process continues over a long period in an apparently inexhaustible fashion.

GICs appear to have anticariogenic properties and may be used for the treatment of early carious lesions and for preventing recurrent caries (Wilson et al 1985, Hicks et al 1986 & Forsten 1990).

Mjör (1996) in a preliminary report, found that a survey from general dental practices showed the clinical diagnosis of secondary (recurrent) caries as the most common reason
for glass ionomer cement replacement. However, this report did not identify the type and brand of glass ionomer material used. It did not discuss the handling of the materials used or the reason for placement of the restoration. The high incidence of clinical diagnosis of secondary caries was mainly detected in three out of the four groups, consequently operator variability may also be possible. More recently Qvist et al (1997) confirmed the cariostatic effects of GICs in a prospective study comparing amalgam and GICs in primary teeth.

5) Erosion:

Initially there was concern over the erosion resistance of the GICs, because of its similarity in chemistry to the silicate cement, but many clinical trials have reported the GIC to be the most durable of all the acid base reaction cements (Mitchem & Gronas 1978, Sidler & Strub 1983, Mesu & Reedjik 1983, Thuniers 1984 & Pluim 1985). This is as result of the polymeric nature of the anion. The active carboxylate groups are connected by covalent linkages impervious to acid attack and the crosslinks are ionic and need to be broken before the matrix can decompose. These findings have been confirmed by other research workers (Crisp et al 1980, Beech & Bandopadhyay 1983, Matsuya et al 1984 & Walls et al 1985).

Setchell et al (1985) reported that the erosion rate was dependent upon the cement type. They suggested that luting cements erode less than restorative cements from the same manufacturer and that cements based on polyacrylic acid erode less than materials based on polymaleic acid.

A later study (Billington et al 1992) has shown that a reduction in the glass particles size alone can dramatically lower the erosion rate. This is attributable to an increase in the
glass surface area, which increases the rate of ion release. It seems likely that it is the cross-linking ions, most probably aluminium ions, that controls the erosion behaviour. These findings also indicate that the erosion occurs within the matrix structure, because a decrease in particle size leads to a reduction in the quantity of matrix. The erosion rates of 3 different types of the cement (restorative, luting and reinforced cements) from the same manufacturer were different (Billington et al 1992).

6) Biocompatibility:

GICs are therapeutic materials and have been demonstrated to have adequate biocompatibility for dental applications (Williams et al 1993). The most significant factor is the selection of a suitable type of GICs for a particular application. Three main factors may influence the biocompatibility of GICs (Nicholson 1991):

- The heat evolved during the earliest stages of the setting of GICs. However, there is unlikely to be thermal damage because very low exotherm values are measured.
- The presence of aqueous polymeric acid in the setting cements. These are relatively weak with a pH of 4.5-5, and the initial step in the neutralisation reaction is so rapid, that the amount of free carboxylic acid present is reduced very early in the setting process.
- The substances leached from the set cement. Aluminium is the only species of doubtful acceptability and it is released at a very low levels (Crisp et al 1980, Brookman et al 1986 & Meryon & Jakeman 1987). Kawahara et al (1979) and Pameijer et al (1981) showed that conventional GIC (luting cement) inhibits cellular proliferation, but is not cytotoxic. It exhibits a greater inflammatory response than zinc oxide-eugenol, but generally less than zinc phosphate cement (Tobias et al 1978, Kawahara et al 1979 & Pameijer et al 1981). Therefore, it is prudent to place a small quantity of protective
cement e.g. calcium hydroxide where less than 1 mm of sound dentine remains over the pulp. A number of causes of post-operative sensitivity to luting cement base have been reported, but it has also been shown that postoperative sensitivity was related to technique rather than to the chemistry of GIC (Wilson 1989).

Sasanaluckit et al (1993) evaluated the biocompatibility of a number of GICs in *in vitro* cell culture studies. Their results demonstrated that there was evidence of cytotoxic effects (cell necrosis and multinucleate giant cells), close to Ketac Silver at 4 days. After 8 days the effect was reduced and it was likely that the extractant was no longer released.

7) Translucency:

The opacity of a GIC can be affected by phase separation of the glass and variations in glass particle size or distribution which lead to a mismatch between the refractive indices of the filler and matrix (Wilson 1989). Modern GICs use clear glass consequently GICs have a closer translucency when matched to tooth enamel.

Knibbs et al (1986) showed in a three year study of Class III GIC restorations that none were in need of replacement as a result of a poor colour match. However, it should be noted that in the original material maximum translucency was only attained after 24 hours because of the slow hydration of the cement. These materials have long-term aesthetic advantages over composite resin as they are more resistant to staining and so colour match is maintained (Wilson & McLean 1988).

2.3.7 Historical development of reinforced GIC:

The GICs development has been through two phases. Initially, attempts were made to enhance the mechanical and handling properties of the cement sufficiently to make
them appropriate for use in the anterior sextant of the mouth. Second, properties were modified in order to extend its range of application to a number of areas including use in the posterior sextants of the mouth.


In an attempt to overcome the lack of strength, Prosser et al (1986) incorporated metal fibres and flakes into the setting cement. Whilst the flexural strength was greatly improved, the system still showed poor wear resistance and now had an enhanced tendency to fracture (Prosser et al 1986).

Seed and Wilson (1978) investigated the effect of incorporating metal fibres or powders into the glass powder, and found that the flexural strength was increased but abrasion resistance was poor which was probably because of a lack of strong bonding between the metal filler and polyacrylate matrix.

Although this early use of metal was unsuccessful, two different methods have now been developed for reinforcing glass ionomer cement commercially available:

a) Adding a metal/alloy to the glass ionomer cement formulation.

b) Sintering metal to the glass particles.

The first development was the inclusion of silver amalgam alloy powder (from normal amalgam alloy) which is directly mixed with the glass fraction of a GIC at level determined by the user, known as Silver Alloy Admix, to give the so-called Miracle Mix, (G-C International) (Simmons 1983). A composition which has been used successfully is approximately 53% m:m of amalgam alloy (El-Mallakh & Sarkar 1990).
The second development was the invention of the silver cermet. Glass cermet cements are made from a glass where a high density of metal powder has been sintered onto the particles. This glass can be made to react with acids to form a cement (McLean & Gasser 1985 & Wasson 1993). The "glass" is obtained by mixing silver in equal volumes with a reactive glass powder, compressing the mixture in a pelletiser and fusing the system at 800 °C, thus the metal becomes sintered onto the glass powder forming the "cermet" powder (McLean 1990). The ratio of glass to silver is fixed at 60:40 (m:m) (McLean & Gasser 1985). This process was used in the production of both an encapsulated material (Ketac Silver) and a hand mixed version (Chelon Silver).

Different metal powders were evaluated including alloys of silver and tin, pure silver, gold, titanium and palladium. Gold and silver were found to be superior in forming a suitable bond with the aluminosilicate glass. Due to the high-price of gold, silver was preferred and good results were obtained using silver powder of average particle size 3.5 μm. The colour of the restoration was further upgraded by incorporating up to 5% by weight of titanium dioxide in the glass powder (McLean & Gasser 1985). However, Williams et al (1992a) noted that the method of fusing the metal to the glass particles rather than by simple addition did not produce any advantages in the strength of the cement.

Williams and coworkers (1992a) also determined that the strength of the modified GIC varied with different loading of metal additions. Therefore, the optimal metal loading level must be established.

Sarkar (1996) has proposed that in reinforced materials, the interaction between alloy particles and matrix (interfacial bonding) is critical for efficient transfer of stress from
matrix to the reinforcement. He found that interfacial bonding so crucial to the performance of reinforced material is lacking in Miracle Mix and cermets. However, no other works have established any significance.

Kerby and Bleiholder (1991) formulated an experimental material by blending stainless-steel powder with glass ionomer cement. The stainless-steel powder was first acid-treated and then added to the glass powder at specific weight ratio 0.5:1 g. They found that the experimental stainless-steel-reinforced cement appeared to be significantly stronger in both tension and compression than conventional GIC.

**Advantages of Miracle Mix:**

The reported benefits of the Miracle Mix formulation were an improvement in the physical properties of the material. The creep resistance was improved and flexural strength increased. The tensile and compressive strengths were also increased. The cement was radiopaque and still had cariostatic properties (Simmons 1983). De Schepper et al (1991) showed that it released a high amount of fluoride. It has been reported that Miracle Mix showed improvements in both tensile strength and creep resistance (El-Mallakh et al 1987).

**Disadvantages of Miracle Mix:**

Miracle Mix cements showed a number of limitations, McLean (1988) has shown that simple mixtures of metal powders and glass failed to form an adequate bond to enamel and dentine. Miracle Mix did not show an improvement in abrasion resistance when compared with the conventional GIC (De Schepper et al 1991). The highest levels
of silver and copper leached from GICs were found for Miracle Mix (53% m:m) when material immersed in liquid after shorter maturation times (Williams et al 1997).

**Advantages of cermets:**

A number of clinical uses have been reported for cermet material, including bases, class V, class I, early class II carious lesions, sealants and repair of castings (McLean & Gasser 1985). It was considered to be a useful alternative to amalgam in the primary molar dentition (Croll & Phillips 1986, Hickel et al 1989, Forsten & Karjalainen 1990). It seems to have much greater wear resistance than conventional glass ionomers (McKinney et al 1985 & 1986, Moore et al 1985 & McLean 1990). The average value of wear of eight hours maturation cermet, obtained with the ACTA wear machine, was 95 µm while that for conventional GIC (Fuji Cap II) was 128 µm (De Gee et al 1996). It has proved most successful in restoring Class II lesion using the tunnel technique when the cement is protected from high occlusal stress in primary teeth (Croll 1988) and in permanent teeth (Croll 1988a). The primary indication for the use of the cermets was for crown build-up, where moderate strength is required, by using normal retentive features (pins, slots, grooves) (C.R.A. News letter 1983). It is also used as a final restoration following a pulpotomy if the tooth is expected to be exfoliated within 2 years (McLean & Gasser 1985). If cermets are used properly, within their limitations, they can be excellent dentine and enamel replacement restorative materials that can last at least six years in the mouth (Croll & Phillips 1991). The set cement could be burnished or polished to produce a smooth surface with metal finish (silver) which enhance the erosion resistance. The incorporated metal gives radiopacity to the cermet which enables the separate identification of restoration and recurrent caries (McLean & Gasser 1985).

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Disadvantages of cermet:

It has been reported that the cermet has a number of disadvantages. Walls et al (1987) suggested that the incorporation of silver reduced the flexural strength and modulus of elasticity of the cermet. The cermet strength was still found to be inadequate for use in high stress bearing areas (Marker et al 1985) and showed a lower creep resistance (El-Mallakh et al 1987). It has also been considered that the cermet was unsuitable for use as a fissure sealant or for restoring occlusal surfaces, because it is difficult to handle and shows evidence of surface cracking and crazing (Smales et al 1990, Wilkie et al 1993 & Lidums et al 1993). Cermets showed poor fracture resistance and wore away when used as a pit and fissure sealant (Croll & Phillips 1991). Forss et al (1991) showed also that the wear resistance of cermet decreased due to hydration when compared to conventional GIC. Cermets have relatively weak bond strength to the enamel and dentine surfaces (Smales et al 1990). It has also been shown that the cermet does not prevent marginal leakage and can not be recommended due to its lack of adhesion to enamel (Gordon-Veitch 1987, Guelmann et al 1989, Jones et al 1990 & Hirschfeld 1992). Although cermets were claimed, by their inventors, to possess a cariostatic action (McLean 1984), fluoride release from the cermet has been shown to be considerably less than conventional GIC (El-Mallakh et al 1990, Forss & Seppa 1990 & Garcia & Chan 1991). The fact that cermets release significantly less fluoride is not surprising, since silver powder replaces 40% by weight of glass powder in cermet and silver also covers the surface of the glass particles. This will inhibit the fluoride release (McLean & Gasser 1985). McDonald et al (1986) have shown that the materials were difficult to adapt to the cavity walls and margins, tended to adhere to metal instruments and were difficult to carve. Thus they concluded that further development of the material and technique was
necessary before it could be recommended for general use in practice.

2.3.8 Material presentation:

The setting reaction of the GIC starts when the components are combined, consequently it is not possible to present the cement in a premixed form. Instead glass ionomer cements are presented to the clinician as two separate components, powder and liquid for manual mixing or alternatively as a capsule which is then mechanically vibrated to achieve a mix.

Hand mixed materials:

GICs are frequently provided as a powder/liquid system. It is well recognised that achieving the correct powder to liquid ratio from a hand mixed/dispensed system could be a problem. The failure to achieve the manufacturers’ recommended powder/liquid ratios can lead to a dramatic decrease in performance (Billington et al 1990). Care must therefore be exercised to ensure that the correct amount of powder is mixed with a given volume of liquid. Hand mixed may lead sometimes to the incorporation of air in the mixed cement.

Encapsulated Materials:

The pre-dosed capsules are now a common form of packaging of commercially available glass ionomer cements. These materials appear to offer the operator some control over the working and setting time. The manufacturers give a range of mixing times for these encapsulated materials and report that increasing the mechanical mixing will reduce the setting time. However, Gee and Pearson (1993) investigated the effect of
mixing speeds on mechanical properties of encapsulated GICs, and found that increasing the speed of mix did not produce the expected reduction in the working and setting time of any of the materials tested. Furthermore, Kilpatrick et al (1994) in their study using Differential Thermal Analysis (DTA) to monitor the thermodynamics of the setting reaction, found that increasing the mixing time of encapsulated glass ionomer cements had no significant effect on the setting time of these materials. However, they found that cold storage of the capsules produced a measurable increase in working time in all the materials.

The factors which have been shown to affect a material’s performance are the speed and pattern of oscillation of the mechanical mixer. Alterations to these two parameters results in variation in the mechanical properties of dental amalgam (Darvell 1981). In a similar study, Gee and Pearson (1993) found that the early compressive strength of one material (Ketac-Fil) increased as the speed of mixing increased. The variation in mixing speed did not appear to affect the early strength of the other two materials (Chemfil & Opusfil) under test and the strength of none of the materials were affected at seven days.

There are two different capsule designs for GICs. One of them only provides a method of mixing. The mixed cement paste must then be scooped from the capsule. The second type contains a nozzle through which the material may be injected into the cavity.
Disadvantages of encapsulated material:

1) The liquid contains lower molecular weight polyacrylic acid to ensure adequate mixing.

2) It is essential that the powder/liquid ratio be reduced if the paste is to be injected from the capsule (Kilpatrick et al 1994).

3) The liquid is quite viscous and it is necessary to keep the pressure on the activator for 2 to 3 second to ensure that all the liquid is expelled from the pillow.

4) The syringable type of capsule can have a small amounts of powder which is either trapped in the nozzle or adjacent to the pillow which is then not incorporated into the mixing (Gee & Pearson 1993).

5) Different types of mechanical mixer may influence the mechanical properties of the encapsulated materials, due to speed and amplitude of oscillation.

6) Variation in powder/liquid ratio of capsules.
2.4 Materials testing:

The ideal *in vitro* strength test for a brittle dental material should have direct clinical relevance whilst being entirely reproducible in a number of different test centres (McCabe et al 1990). Comparisons between published data are difficult to make owing to the variety of materials, and test methods employed (Piddock et al 1987 & Billington et al 1990).

To make test results as clinically relevant as possible, it is advantageous to reproduce as many features similar to the oral conditions in the test method. This includes the production of test samples of similar shape and dimension to those found clinically, and then testing them to failure in a manner similar to the predicted or known modes of clinical failure. Test specimens should also be stored under conditions representative of those found in the oral cavity.

Mechanical properties:

A material’s mechanical properties such as predicting behaviour under load will provide some understanding, but it is also important to recognise that no single mechanical property can give a true measure of a material’s quality. It is also essential to understand the forces experienced in clinical use and the principles involved in the variety of mechanical tests performed.

Strength and rigidity are necessary where occlusal stresses are placed on a restoration. Farah et al (1975) have shown that the fracture resistance of an amalgam restoration decreases as the modulus of elasticity of the base material is lowered. Knowledge of the strength factors of a particular material assist the clinician in selecting the appropriate material for a particular application.
2.4.1 Compressive strength:

The measurement of compressive strength is technique sensitive. It appears to produce results dependant upon the methods of specimen preparation, the components of the specimens, chemistry, operator and laboratory variables (Williams et al 1992a). McCabe et al (1990) investigated mechanical testing of restorative materials and concluded that compressive strength is not a suitable test for inclusion in International Standard Testing in view of the tremendous variability in the results obtained. They also concluded that it has no fundamental meaning for a material can only fail either by the separation of planes of atoms i.e. tensile failure or by the slipping of planes of atoms i.e. shear failure. However, compressive strength is widely used as the criterion of strength of glass ionomer cement despite the difficulty in interpretation of the findings. It is usually considered that the cylinder should have a length approximately twice that of the diameter (Craig 1989) to avoid force distribution complication as a result of overlapping force vectors in the length of the cylinder when it is too short. Manufacturers of commercial GICs often quote values for the compressive strength of their materials without specifying how these values have been obtained. This is a problem because the values obtained often depend upon the conditions used such as specimen dimensions, nature of the material and the rate of loading.

One study found that the compressive strength test was the least discriminatory test between materials due to a wide scatter in the results (Williams et al 1992a). However, compression force has some importance in the process of mastication.

The standard method for testing compressive strength for GIC is described in ISO 9917 (1991). In this method cylindrical specimens (6 mm x 4 mm in diameter) are prepared and loaded in compression.
The small size specimen is used for the following reasons:-

1- The effect of specimen size upon compressive strength of GIC has been investigated by Iguodala et al (1991) and no significant differences were found in the values obtained for the differently-sized specimens. However, the minimum height/diameter (h/d) ratio must be maintained.

2- The specimen is as close as possible to a clinically relevant size.

3- Economic.

4- It is suitable for preparing specimens, using capsulated or fast setting materials, where one capsule will normally produce sufficient material for each specimen and no double mixing is required.

2.4.2 Flexural strength:

According to Prosser et al (1986) the flexural strength test is the most appropriate measure of the strength of a dental restorative material and offers the best practical and most reliable estimate of the strength.

The flexural strength test is a collective measurement of a number of types of stress simultaneously. It can be regarded as a representation of the type of forces generated in the oral cavity. The occlusal surface of a restoration is under compression and the base of the restoration is under tension.

1) The uni-axial flexural strength:

Three and four point flexure tests have been used widely for strength evaluation of single component brittle materials (Bryant & Mahler 1986 & Soderholm 1986).

Three and four point beam testing are accepted methods of establishing tensile strength.
A standardised 'beam' of material is prepared, and then loaded at either one or two points on one side, whilst being supported at either end, on the other side (Edwards et al 1983).

The limitation of uniaxial flexural tests are:-

1- Undesirable edge fracture can increase the variation of the failure stress value.

2- These methods were designed for engineering and are usually associated with large specimens.

3- Construction of large specimens is not usually convenient for brittle dental materials.

4- Suitable quantities of dental restorative materials are not often available to prepare a sufficient number of specimens for assessment of statistically significant differences.

5- The residual stress set-up during the setting reaction and the flaw characteristics induced in large specimens may not be representative of those that are present in smaller clinical restorations (Ban & Anusavice 1990).

6- The specimen preparation is difficult and any marginal irregularities or defects within the bulk of the specimen, will influence the results and may also be difficult to detect visually (Edwards et al 1983 & Ban & Anusavice 1990).

The four point bending test assembly, as shown in the Fig 2, is a simple beam shape, 22 mm long x 2 mm (cross section), supported by rollers set 20 mm apart in the lower span while the distance between the upper span rollers was 6 mm. The load is applied to the specimen area between the two rollers of the upper span. The mode of loading at all four points is compression while between the two lower span rollers, a force in tension is setup.
The difference between four and three point bending tests is the loading pattern, at the mid point of the specimen for the three point test rather than at an area between two rollers for the four point. The three point has also three compression mode points rather than four. The four point application is to an area, to ensure that the forces to be well distributed and prevent specimens distortion or failure along a fault line.

2) The Bi-axial flexure strength:

The measurement of the strength of brittle materials under bi-axial flexure conditions is often considered more reliable than uni-axial flexure because:-

1- The maximum tensile stresses occur within the central loading area.

2- The stress distribution eliminates any edge discrepancies in the specimen to affect the
results, since the annular ring on which the disc rests defines the margin of the specimen.

3- The disc’s surface/volume ratio compares well with that of clinical restorations.

4- No surfacing of the specimens is required.

A wide variety of loading arrangements have been developed for bi-axial flexure strength:-


3- Ring-on-balls (Shetty et al 1983).

The bi-axial flexural strength test, the Ball-on-ring method, (shell test), by Wilshaw (1968) has been used frequently and recommended for the determination of fracture characteristics of brittle materials. It is because a ball-on-ring method is simpler and provides a better simulation of clinically relevant sample size than that used for the other methods (Williams et al 1992a).

The shell test involves the fabrication of thin discs of material which are then placed individually on an annular knife edge. They are then loaded in the centre of the specimen, by a ball-ended indentor until failure. The original theory on which the test is based was devised by Timoshenko & Woinowsky-Kreiger (1959). More recent advocates of this method include Sced (1977), Piddock et al (1987), Ban and Anusavice (1990) and Williams et al (1992a).

Other mechanical tests such as diametral tensile strength, hardness and fracture toughness are possible but have not been considered in this project because of the time
limit for this project and its focus on more clinically relevant tests.

2.4.3 Evaluation of adhesion to enamel and dentine:

The majority of investigations on adhesion to dentine have been conducted in vitro and as such, the results should be treated with caution. This is because additional variables such as dentinal fluid, intratubular pressure, placement technique, tooth age and vitality, environmental conditions, masticatory forces, thermocycling and microleakage all influence the bond strength of the materials in vivo (Pashley & Depew 1986 and Soderholm 1991). However, time is required to establish trials and for analysis of subsequent results in vivo, so great reliance is placed on laboratory testing prior to the launch of commercial materials.

The most common bond strength tests for adhesion are tensile and shear tests. The direct uniaxial tensile test requires a universal joint system to align the specimen and substrate so that the forces act at right angles away from the specimen surface. However, stress concentrations, peel and shear forces can occur during this alignment, providing false results.

In shear testing, the force is applied parallel to the substrate as shown in Fig 6. Care must be taken to avoid peeling of the bond and to ensure that the bond fails at the plane surface determined by the test, and not in the mode created by characteristics of the materials. This may explain the often small scatter of results with shear in comparison with tensile tests (Rees & Jacobson 1990 & Kerby 1992).

Other tests for bond strength include the blister test, where increasing air pressure
is applied to the bond until rupture occurs via a hole drilled through the substrate (Williams et al 1973), the centrifugal test, where the specimen is spun at increasing speeds until peripheral tensile forces rupture the bond, has also been used (Hotz et al 1977). In the latter test, the specimen is likely to be subjected to peel and shear forces created by the acceleration from rest to the point of bond failure. However, due to practical difficulties, the last two tests have been rarely used in dental research.

Previously, tensile bond strength was considered to be the preferred method of adhesion evaluation for dental materials, but more recently shear test has been considered to be more predictive of intra oral performance (Retief 1991 & Yap et al 1994).

2.4.4 Erosion resistance: (Acid resistance).

Dental erosion is the progressive loss of hard dental tissues or a restoration by a chemical process not involving bacterial action (Watson and Tulloch 1985).

The solubility of dental cements, (which implies a simultaneous degree of erosion), may be determined clinically or in the laboratory. Clinical tests are difficult since they require a year or more to show significant changes, and variations in oral conditions among trial subjects are likely to lead to scatter in the results. For these reasons, laboratory trials have long been recommended as a useful short-term source of information on the behaviour of the material.

The classical laboratory test is based on the American Dental Association Specification Test Number 9 (ADA 1963) in which a disc of cement is immersed in water for 24 hours after which the storage water is evaporated to dryness and the weight of eluate thereby measured. This test has been criticised, because it reflects only an initial leach-out
process which is often followed by a much slower dissolution, the rate of which is unrelated to the initial process (Kuhn et al 1984). It also contains no element representing the mechanical erosive action which undoubtedly occurs at certain point in the mouth (McCabe 1982). This test is also inadequate for cements where the liquid is part of the component (water and resin based cements). The water and monomer may be released and then evaporate, therefore, false results may be achieved.


The jet-test uses a volume (usually around 6 litres) of dilute lactic acid (0.02 M) which is circulated through the apparatus by a pump. A number of cement samples are arranged in small holders and the liquid emerging from a series of small jets impinges on the cement samples, one beneath each jet. In this way, a mechanically erosive element is combined with the chemical erosion which occurs during the test procedure. As a dilute acid, rather than water, is used as the test solution, the erosion proceeds in hours rather than days. After a comparatively short time, the erosive wear of the cements is indicated by cement loss. These can be measured either by weight loss or loss of height of the specimen.

The jet test has the advantage over the simple immersion solubility test carried out in static distilled water in that the erosion rates from the jet test rank the different chemical classes of dental cement in the same order as their dissolution rates in vivo (Mitchem & Gronas 1978, Pluim et al 1984 & Wilson & McLean 1988).
Williams et al (1994) evaluated some factors influencing the lactic acid jet test and found that the test temperature would more appropriately be increased from 24 °C to 37 °C to approximate to the condition in the oral environment and minimise temperature effects due to heating by the recirculating pump. It is also easier to control the liquid temperature, normally raised during circulation by the pump, at 37 °C than at 24 °C. Erosion may be measured by weight loss, which is easier to perform, is an average measurement and does not rely upon a depth measurement on an unevenly eroded surface. However, the weight loss measurements of different density of many dental materials lead to substantial difference in loss of material, although weight loss may be similar. This is particularly the case with a metal reinforced cement. Therefore the use of both measurements will confirm the results.

2.4.5 Microleakage:

Microleakage may be defined as the clinically undetectable passage of bacteria, fluids, molecules or ions between a cavity wall and the restorative material applied to it (Kidd 1976 & Trowbridge 1987). Microleakage at the tooth/restoration interface is considered to be a major factor influencing the longevity of dental restorations and can lead to marginal discolouration, marginal breakdown, secondary caries and post-operative sensitivity (Alani & Toh 1997).

Detection of microleakage:

Many techniques have been developed to assess the marginal adaptation of dental restorations both in vitro and in vivo and to evaluate the leakage at the interface between tooth and restoration.
The in vitro techniques include the use of dyes, bacteria, chemical and radioactive tracers, air pressure, light microscopy, scanning electron microscopy, artificial caries media, neutron activation analysis, electrical conductivity and the influence of both thermal and mechanical cycling (Shortall 1982, Taylor & Lynch 1992 and Alani & Toh 1997).

The dye-penetration method is the oldest and the most commonly used method of detecting leakage in vitro. It involves placing an extracted, restored tooth in a dye solution for a predetermined time. This is followed by washing and sectioning of the specimen and then examination of the sections under magnification to determine the extent of leakage around the tooth-restoration interface (Going 1972).

Glyn Jones et al (1988) have used image analysis technique to measure the length of marginal leakage. However, this technique is prone to subjective problems. It was also suggested that leakage assessment based solely on the leakage length was inadequate to allow differentiation between spaces at the tooth/restoration of various size (Youngson et al 1990).

Advantages of dye penetration method:
This method can:-
1) Allow the production of sections showing leakage in contrasting colours for both tooth and restoration materials.
2) Take place with no further chemical reaction.
3) Take place without the need for exposure to potentially hazardous radiation used in radio-isotope studies.
Disadvantages of dye penetration method:

The disadvantages of this method are that it is:-

1) a qualitative method of assessment, although often carried out by more than one examiner.

2) a destructive technique, assessment around the restoration as a whole is difficult as information other than in the plane of section is not available.

Going (1972) suggested that fluorescent dyes are useful in showing microleakage because they are detectable in dilute concentrations, sensitive to ultraviolet light and easy to photograph as they contrast sharply with natural fluorescence of teeth whilst being non-toxic. Leinfelder et al (1986) and Bullard et al (1988) have established that a 0.5% solution of basic fuchsin dye is effective in determining the extent of microleakage present under restorations. Other dyes commonly used are methylene blue and neutral red dye.

Mechanical cycling of restored teeth may result in the production of marginal gaps, either permanently or only while the tooth is under stress (Jorgensen et al 1976). Repeated thermal and/or mechanical cycling may be used to accelerate leakage and so obtain results in a reasonable time whilst simulating the intraoral condition (Rees & Jacobson 1990).

Tong and Herrin (1987) found that load-cycled and thermo-cycled Class II composite restorations exhibited a greater gap at the resin-dentine interface than uncycled controls. Crim & Garcia-Godoy (1987) showed that short-term cycling appeared to be as effective in demonstrating marginal leakage as protracted cycling regimens.

Chewing cycles and movement during chewing has been investigated in terms of time
Neill (1967) has studied the nature and frequency of the occlusal contacts occurring between opposing complete dentures and found that the average chewing rate was about 80 chews per minute. Schwartz et al (1984) found that age and sex did not affect time, number of cycles, or the time/cycle ratio, but the measurements were strongly affected by the type of food being eaten.

There appear to be no papers in the literature which specifically addressed the number of chewing cycles of a normal adult person per day. However, Delong and Douglas (1991) have integrated two servo-hydraulic actuators operating under close-loop control to produce a very accurate physiologic representation of the force-movement cycle of human mastication. The system operating at 4Hz, can reproduce the force pattern of human mastication. They assumed that 250,000 cycles in the artificial mouth equals one year in the human mouth. Delong et al (1985) found that the wear of amalgam in the artificial mouth gave a linear relationship in respect of the vertical loss. Furthermore, Delong et al (1986) found that the volume of occlusal material lost as a result of wear increased as a direct functional of time. Delong et al (1992) found that the relationship between volume of material removed and number of cycles was linear. This is in agreement with other works (Coffey et al 1985, Delong et al 1989).

Other techniques:

Bacteria have been used in the detection of microleakage. This is achieved by placing restored teeth in broth cultures. The filling materials were subsequently removed and dentine shavings from the base of the cavity cultured. This provided purely qualitative results depending mainly upon the presence or absence of bacteria in part of the dentine shavings examined (Ross et al 1955 & Seltzer 1955).
The chemical tracer method uses silver ion in the form of silver nitrate solution. The silver ion is extremely small when compared to the size of a typical bacterium (0.5-1 \( \mu \text{m} \)) and thus is likely to penetrate further. Soderholm (1991) suggested that chemicals equal to the molecular sizes of bacterial toxin should be used. Wu et al (1983) suggested that staining with silver nitrate produces an accurate evaluation of microleakage and specimens can be stored, because the particles of silver precipitate and will not diffuse into adjacent tooth structure. However, there are difficulties in interpretation of the results.

Long term monitoring of microleakage can be achieved by using radioactive tracers. The use of isotopes such as calcium in the form of calcium chloride permits detection of minute amount of leakage. This involves the use of extracted restored teeth and the immersion of the specimens in a solution of the isotope for several hours. Then autoradiographs of the cut surfaces are taken to detect the tracer (Going 1972 & Alani 1997). However, it lacks clinical relevance (Powis et al 1988), and is quite complex and costly (Alani 1990).

The air pressure technique involves passing compressed air through the root canal of restored tooth into the interspace and examines the restoration under water. The critical pressure at which a bubble forms at the tooth surface is measured and recorded against time (Granath & Svensson 1970). Microscopic examination of the submerged restoration for the release of air bubbles from the margins provides a subjective view of the margin seal. The main limiting factor of this method is that it does not simulate clinical conditions.
Light microscopy and scanning electron microscopy (SEM) are used in leakage detection. The light microscopy has a limit of resolution, however, and requires a plane surface at the interface for accurate measurements. Examination by SEM provides direct visual observation of the adaptation of restorative materials to cavity margins. This method requires replication using elastomeric impression materials to make a negative and epoxy resin to form the positive. This technique is useful for the intraoral study of marginal adaptation but is not frequently used (Rees & Jacobson 1990), difficult to quantify and may introduce errors related to drying, cracking, distortion and sectioning particularly if replication is not used (Kidd 1976).

Artificial caries-like lesions are produced in vitro using either bacterial cultures or a chemical system (acidified gelatin gel) technique. These produce artificial lesions around the restoration. The incidence of leakage is observed by sectioning the tooth and restoration (Kidd 1978).

Neutron activation analysis involves immersing the restored tooth in an aqueous solution of a nonradioactive manganese salt. All the salt adhering to the outside of the tooth is then removed and the tooth placed in the core of a nuclear reactor. This results in the nonradioactive manganese salt being activated and the x-ray emission can then be measured (Going et al 1968). This method has the advantage that results can be quantified, but it is complex and costly (Going 1972).

The conductimetric technique is to evaluate changes in the dimensions of the cavity wall/restoration interspace using an electrochemical cell. It involves immersion of
the restoration in a bath of electrolyte, and the cavity wall/material interface is incorporated into an electric circuit. As the interspace fills with electrolyte so the resistance of the circuit decreases. A potential/time curve could then demonstrate the variations in interspace dimensions (Jacobson & Van Fraunholfer 1975). However, there are problems in avoiding the conduction of current by the electrolytes in dentine. This method has also proved to be sensitive and quantifiable and seems to lack clinical realism (Shortall 1982).

Trowbridge (1987) reported that most in vitro investigations fail to simulate intraoral conditions. Problems such as poor correlation between assessment conditions, usage of extracted teeth and susceptibility of the tooth/restoration interface to dimensional changes during experimental manipulation continue to exist.
2.5 Clinical trials:

Clinical studies are relatively limited in numbers compared to the large number of laboratory studies (Duke 1992). Most studies are carried out under ideal conditions by a single dentist. These studies may also be undertaken in dental hospitals using patients including dental students or well motivated patients. These do not represent the typical patient in a General Dental Practice. Since there are large differences in operating techniques for individual dentists, such studies merely demonstrate that the investigator is able to achieve a certain result with restorations of the test materials being used under a standard set of conditions (Roulet 1994).

The design of clinical studies to evaluate posterior restorative materials is further complicated by the influence of a number of clinical variables, such as the tooth to be restored, the size and complexity of the restoration and the presence and nature of occlusal contacts. These have all been identified as affecting the behaviour of the material.

2.5.1 Clinical trial variables:

The major variables which could complicate clinical trials designed for evaluation of restorations in class I and II sites (Bayne et al 1987), have been reported as:-

1- Operator.
2- Material.
3- Intra-oral site.
4- Patient.
5- Poor precharacterization.
Operator:

Operator skills differ widely in general dental practice, dental hospitals and other health care institutions. The wide variations in operator techniques may affect the outcome of a clinical study. The operator can influence restoration design/outline and also placement and finishing techniques, if more than one operator is used. Each operator should place an approximate equal number of restorations of each materials to avoid undue bias as a result of idiosyncratic behaviour of one operator (Osborne & Gale 1974 & Mahler & Marantz 1980).

Material:

The influence of materials on clinical trials is well documented especially with regard to amalgam and composites studies. Material factors include formulation aspects which may vary due to manipulation effects and changes in composition and degradation during the life span of a restoration (Bayne 1992).

Osborne et al (1991) found that amalgam alloy brand had an influence on the survival of the restoration after eight years. High copper amalgam restorations, in general, exhibited less deterioration than the conventional alloys (Jacobsen 1984a & Letzel et al 1989).

Intra-oral site:

The tooth type, position and the restoration type have had a great influence on the design of clinical evaluations of restorations. A similar number of the tooth type should be scheduled for each material being tested. The contra-lateral pairing of restorations is one method which has been suggested for controlling the influence of tooth type and position. Goldberg et al (1979) showed in their study that the tooth type and the number
of restored surface had a significant effect on clinical trial design. They found that restorations in molars showed more fractures than those in premolars and in particular mandibular premolars showed the least.

**Patient:**

The specific combination of the oral environment and occlusal loading has been shown to have a significant effect on restorations. The contribution due to patient should also be considered, both with respect to the size of the patient sample and the distribution of the materials within each patient (multiple restoration including the control). The objectives of such measures are to ensure an unbiased example over extremes in the oral environments (Mahler & Marantz 1979). In a cross sectional clinical evaluation, Goldberg et al (1981) concluded that oral hygiene variation had an effect on the probability of secondary caries with moderate marginal deterioration.

**Poor precharacterization:**

Some investigators are emphasising the importance of much more extensive precharacterization of the site of the restoration. Bayne et al (1991) have noted the origin of the site being restored. The physiologic age and pulp status need to be estimated so that change or particular predispositions can be documented. The occlusal status and bruxing levels appear to influence the retention and potential sensitivity associated with restorations (Heyman et al 1991).

**2.5.2 Evaluation of occlusal wear:**

Current methods of clinical evaluation of occlusal wear still remain highly
subjective, involve standard clinical diagnostic equipment and require a lengthy evaluation period. There is now an increasing demand for more accurate and reliable methods. Attempts to achieve these have been directed, principally towards the standardisation of clinical judgements, by the provision of various systems of guidelines of criteria upon which to base them. The efficiency of using the particular criteria selected are themselves open to questioning (Smales 1983).

Gruebbel (1950) has designed four categories of fault but gave no details of the examination procedure. He also failed to discuss inter-examiner variability. The faults for evaluation which were proposed were:-

1) marginal discrepancies.
2) poor restoration of tooth form.
3) fractured restorations.
4) fractured tooth.

Abramowitz (1966) in the U.S. Public Health Service, conducted an investigation in which two dentists rated restorations as either satisfactory or unsatisfactory.

Hammons and Jamison (1967) defined ten criteria to which the dentist should refer when making the assessment:-

1) anatomical carving.
2) marginal ridge relation; correspond to the height of adjacent teeth.
3) contact; proximal contact with adjacent teeth.
4) contour; the original, natural surface of the tooth.
5) marginal integrity.
6) condensation of the restoration (according to the type of a restoration).
7) occlusion; assessed by use of articulator paper.
8) supporting tissue integrity.
9) post-operative lavage.
10) surface smoothness.

Each criterion was judged to be:-
a) excellent.
b) acceptable.
c) inadequate.

The assessors were independent dentists and were not given any guidelines to differentiate between the categories of excellent and acceptable.

Lotzkar et al (1971) designed a study to evaluate freshly placed amalgam restorations. They defined four criteria:-
1) adaptation to the cavity.
2) contour of the restoration.
3) contact to the adjacent teeth.
4) occlusal contact.

They suggested that the satisfactory restoration had to conform to these criteria. Considerable variation was detected among the individual operators and their assessments.

A different method has been developed by McLean and Short (1969). They divided the physical characteristics of amalgam and resin restoration into three groups:-
1) surface texture and contour.
2) anatomical form.
3) marginal integrity.
Cvar and Ryge (1971) developed what are now known as the U.S. Public Health Service (U.S.P.H.S.) criteria. This system was then considered to be one of the primary methods of assessing the performance of amalgam and the aesthetic restorative materials clinically and continues to be used to the present day. This system assess a variety of criteria for restorative materials (see below). These assessments are made immediately after the restorations are completed. Baseline evaluation is followed by evaluation on a regular basis, frequently annually, performed by two trained dental examiners. To eliminate examiner bias each examiner’s rating is performed independently without the knowledge of the identity of the materials’ previous ratings or other examiners’ ratings. All ratings are collected and entered into a computer which was programmed to identify any disagreement.

2.5.3 Direct evaluation criteria:

The criteria for evaluation were retention, marginal integrity, anatomic form, surface texture, post-operative sensitivity, marginal discolouration and colour match. The last two criteria are utilized to assess the aesthetic restorative materials.

The U.S.P.H.S criteria evaluation system consists of a progressive rating scale where the first two scores of each category, "Alfa" (A) & "Bravo" (B), are considered to be clinically acceptable. The next score "Charlie" (C), represents evaluations which indicate unsatisfactory performance.

The clinical criteria for direct evaluation are as follows:-

Retention:

Alfa (A): The restoration was intact and fully retained.

Bravo (B): The restoration is partially retained with some portion of the
restoration intact.

Charlie (C): Complete loss.

Marginal integrity: (with sharp #6 probe and visually)

Alfa (A): The probe does not catch when drawn across the surface of the restoration toward the tooth or if the explorer does catch, there is no visible crevice along the periphery of the restoration.

Bravo (B): The probe catches and there is visible evidence of a crevice, into which the probe will penetrate or base is visible.

Charlie (C): The probe penetrates into a crevice that is of such depth that the dentine or base is exposed.

Anatomic form:

Alfa (A): The general contour of the restoration follows the overall contour of the tooth.

Bravo (B): The general contour of the restoration does not follow the overall contour of the tooth.

Charlie (C): There is sufficient material lost to expose the dentine or the base.

Surface texture:

Alfa (A): The surface of the restoration is smooth and shiny.

Bravo (B): The surface of the restoration is smooth and dull.

Charlie (C): The surface of the restoration is grainy and rough.
Post-operative sensitivity:

**Alfa (A):** Absent.

**Bravo (B):** Present thermally or digitally induced - short duration 1-4 seconds.

**Charlie (C):** Present thermally or digitally induced - long duration 5-30 seconds.

Sensitivity is assessed by questioning in conjunction with an air blast directed on to the restoration from a distance of 2 cm.

However, the use of subjective criteria methods for clinical research has disadvantages including the need to train and maintain the evaluators at an arbitrary 85% level of concordance.

There were some problems which made the U.S.P.H.S. criteria difficult to use internationally. The small numbers of ratings used within each categories means that small differences within or between various materials cannot be detected (Merill et al 1975 & Osborne et al 1976).

Taylor et al (1989) compared direct and indirect methods for analysis of posterior restorations. They found that the U.S.P.H.S. criteria for anatomic form has been found to be relatively insensitive to the assessment of occlusal wear. The system does not produce quantitative wear measurements and is not sufficiently sensitive to distinguish important early changes in wear behaviour.

2.5.4 Indirect evaluation procedures:

There are several indirect evaluation systems that compare casts of restored teeth to a series of standards exhibiting incremental amounts of occlusal wear.
Leinfelder et al (1983) have employed a method which uses standard casts which are compared with the restoration to be evaluated. A series of die stone casts of composite restorations exhibiting varying degrees of wear selected for evaluation. Addition silicone impressions were then made of the restored occlusal surface. Next the negative replicas were sectioned faciolingually at three different intervals. The distance from the cavosurface margin to the composite surface was determined using a travelling microscope. On the basis of these measurements six different casts were selected as standards. The average wear represented by each cast ranged from 0 to 500 μm.

Three years later, Moffa and Lugassy (1986) developed an indirect evaluation systems (the M-L scale), consists of eighteen cylinders with progressive depressions ranging from 0-1000 μm in depth.

Recently, a system developed by Rheinberger for Ivoclar-Vivadent provided an eighteen category standard scales. It consists of tooth-shaped, tooth-sized dies with restoration-like incremental defects ranging from 25-1000 μm (Bryant 1990). The scales was intended to be read in 25 μm increments from 0 to 200 μm, in 50 μm increments between 200 and 500 μm and 100 μm step from 500 to 1000 μm (1 mm). The indirect assessment of occlusal wear may be evaluated through the use of stone replicas. A polyvinylsiloxane impression material system is used to recorded the study teeth and restorations. The models are poured in stone and stored until assessed by the examiners.

Difference among these systems have reported to be acceptable (Leinfelder et al 1986 & Goldberg et al 1987) and comparatively small when assessments were carried out by experienced evaluators (Bryant 1990). However, the use of Ivoclar-Vivadent standard
cast provided evaluators with the greatest sense of confidence in their assessments and achieved the most uniform set of average wear values (Bryant 1990).

There have been a relatively small number of studies on clinical quality and longevity of glass ionomer restorations, most of them were in primary teeth (Kilpatrick 1993, van Waes 1993 & Kilpatrick 1995), and a limited number were in permanent teeth (Tyas 1991 & Horsted-Bindslev 1994). The clinical quality and longevity of previous comparative work of conventional glass ionomer and amalgam restorations in primary teeth have always shown that amalgam performed better (Fuks et al 1984, Kullmann & Freers 1984, Engelsmann et al 1988, Hickel & Voss 1990, Ostlund et al 1992 & Papathanasiou et al 1994). In most of these studies, the observation period was short and include limited number of restorations. However, clinical studies based on a longer period of time (three and five years) also concluded that one conventional GIC (Ketac-Fil) is not an appropriate alternative to amalgam (Dispersalloy) for class II restorations in primary teeth (Welbury et al 1991 & Qvist et al 1997). Secondary caries was stated as the main reason for replacement of amalgam restorations while the marginal fractures and tooth fractures were most common for the GIC (Ketac-Fil). Therefore, the increasing use of glass ionomer for restorations in the primary dentition may be based largely on dentists' belief in the cariostatic effect of the material due to its prolonged release of fluoride.
Chapter (3)

Materials and Methods
3 Materials and Methods:

3.1 Materials:

The three materials selected for use in this project were as follows:-

a) A metal reinforced GIC restorative material (Hi-Dense), provided in an encapsulated form by the manufacturer (Shofu, Advance Healthcare Ltd, Kent, England, UK).

b) A conventional GIC, an experimental high powder/liquid ratio material (EXPT), provided in an encapsulated form by the same manufacturer.

c) A disperse phase amalgam alloy (Dispersalloy), provided by the manufacturer, (Dentsply Ltd, Addlestone, Weybridge, UK), in self-activating capsules form was included in the mechanical testing and for comparison in clinical use. Details of the compositions of these materials are shown in Table 1.

Table 1: Materials composition: (weight%)

<table>
<thead>
<tr>
<th></th>
<th>Hi-Dense</th>
<th>EXPT High P/L restorative</th>
<th>Dispersalloy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass (calcium fluoro-alumino-silicate)</td>
<td>57%</td>
<td>Glass (calcium fluoro-alumino-silicate) 72%</td>
<td>Silver 70%</td>
</tr>
<tr>
<td>Polyacrylic acid</td>
<td>12%</td>
<td>Polyacrylic acid 16.6%</td>
<td>Tin 16%</td>
</tr>
<tr>
<td>Tartaric acid</td>
<td>1%</td>
<td>Tartaric acid 1.4%</td>
<td>Copper 13%</td>
</tr>
<tr>
<td>water</td>
<td>8%</td>
<td>water 10%</td>
<td>Zinc 1%</td>
</tr>
<tr>
<td>Alloy (Ag:Sn:Cu)</td>
<td>18%</td>
<td></td>
<td>Mercury (Liquid)</td>
</tr>
<tr>
<td>(59.5%) Silver</td>
<td>10.71%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(27.5%) Tin</td>
<td>4.95%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(13%) Copper</td>
<td>2.34%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Titanium Dioxide</td>
<td>4%</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Storage media for the long term mechanical tests was initially in both deionised distilled water and an artificial saliva solution. The artificial saliva formula was originally described by Tanni & Zucchi (1967). The composition of artificial saliva is set out in Table 2.

Table 2: The artificial saliva composition:

<table>
<thead>
<tr>
<th>Material</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dipotassium hydrogen phosphate .... K₂HPO₄</td>
<td>0.20 g</td>
</tr>
<tr>
<td>Calcium phosphate ...................... C₃(PO₄)₂</td>
<td>0.30 g</td>
</tr>
<tr>
<td>Potassium thiocyanate .................. KSCN</td>
<td>0.33 g</td>
</tr>
<tr>
<td>Sodium hydrogen carbonate .............. NaHCO₃</td>
<td>1.50 g</td>
</tr>
<tr>
<td>Sodium chloride ........................ NaCl</td>
<td>0.70 g</td>
</tr>
<tr>
<td>Potassium chloride ..................... KCl</td>
<td>1.30 g</td>
</tr>
<tr>
<td>Urea .................................. (NH₂)₂CO</td>
<td>0.13 g</td>
</tr>
</tbody>
</table>

The preparation of the solution was carried out by weighing the constituents to an accuracy of 0.01g and then dissolving them in one litre of deionised distilled water in a volumetric flask. CO₂ gas was then bubbled through the solution until a pH of 6.7 was reached. The pH of the solution has been monitored throughout the experiment.

3.2 Mixing method:

Both glass ionomer based materials were provided in capsules, Fig 3. Each capsule had an aluminium pillow containing the acid sited in the top. The liquid was dispersed by applying pressure to the capsule ends for 3 seconds. This ruptured the pillow expelling the liquid into the main body of the capsule.
The capsule was then placed in a mechanical mixer (Silamat Plus, Type S 10, Vivadent) at an oscillating speed at 4500 cycle/min for 15 seconds. Dispersalloy was also dispensed in capsules. In this case the pillow contained mercury. Activation was achieved by the presence of a pestle within the capsule which ruptured the pillow as mechanical mixing commenced. The mixing time was also for 15 seconds, as recommended by the manufacturer. Each material was removed from the capsule with a spatula.

3.3 Pilot studies methods:

A number of preliminary experiments were carried out to determine:-

a) the optimum technique for specimens construction.

b) the regime for specimens storage.
c) the level of operator variability.

The manufacturer recommended the use of a glaze in material clinically to prevent changes in the water constituent of the cement. While its use under certain circumstances is essential clinically, it is not clear whether this would be the case in laboratory testing.

3.3.1 Compressive strength:

Pilot Study 1: (Specimen with glaze applied and conditioned for 10 min).

The metal reinforced glass ionomer material (Hi-Dense) was used for this pilot study. Stainless steel split moulds (6 mm x 4 mm internal diameter) were used in the construction of the specimens. The internal surface of each mould was previously coated with a silicone spray (Releasil, Dow Corning Ltd, Barry, Glamorgan, UK) to facilitate the removal of the hardened cement specimen. The materials were mixed according to the manufacturer’s recommendation and packed into the split mould. The mould was filled to excess and then firmly seated on a metal plate. The extruded cement was removed, and a further metal plate placed in position on the top of the mould. The assembly was clamped together and then placed in a thermostatically controlled oven at 37°C for 10 min. The specimens were removed from the mould and checked for voids and chipped edges using a magnifying lens at x8 magnification. Two layers of light cured protective resin (Hi-glaze, Shofu, Advance Healthcare Ltd) were applied on the specimens surface and cured for 20 second with a polymerizing light (EuroMax, DeTrey, Dentsply) operating in the 460-470 nm range. The resin consists of Bis-GMA, the reaction product of bisphenol-A and glycidyl methacrylate, 60%, and triethylene glycol dimethacrylate (TEGDMA), 40%.
Six specimens were constructed, and then stored in deionised distilled water in the oven at 37 °C for 24 hours from the start of mixing. Each specimen was then placed between the plates of a universal load testing machine (Hounsfield H25K, Croydon UK) prior to loading at a cross head-speed of 1 mm/min using a 5000 N load cell. The load at failure was recorded on a strip-chart recorder. The compressive strength was calculated from the formula:-

\[ \text{Compressive strength} = \frac{4 \, P}{\pi \, d^2} \]

Where \( P \) = the load at failure.

\( d \) = the diameter of the cylinder. (ISO 9917 1991)

**Pilot Study 2:** (Specimen with glaze applied and conditioned for 60 min).

The technique and testing method were as described in the previous compressive strength pilot study. The conditioning period of the material within the mould was increased to 60 min, and glaze was again applied prior to storage at 37 °C for 23 hours in water. Eight specimens were constructed.

**Pilot Study 3:** (Specimens which were surface ground with no glaze applied and conditioned for 60 min).

Sixteen specimens were constructed for this part of the program. The technique and testing method were similar to the second study except that no glaze was applied and the ends of the specimens were ground flat before removal from the mould, using abrasive powder (silicon carbide 400 grit) on a flat glass surface. The specimens were removed from the mould after 60 min and stored in deionised distilled water for 23 hours until testing.
Pilot study 4: (Specimens which were surface ground with glaze applied and conditioned for 60 min).

The technique and testing methods were similar to the pilot study 3. However, the light cured resin was applied on the specimens surface before being placed in deionised distilled water. Nine specimens were produced for this study.

3.3.2 Bi-axial strength (shell test):

Pilot Study 1: (Specimens packed between glass slides with glaze applied and conditioned for 60 min).

The metal reinforced glass ionomer cement was again used for specimen construction. A total of six specimens were constructed using a split ring mould (10 mm diameter x 1 mm). The mould was placed on a glass slide, the material was packed to excess and a second glass slide placed on top. Finger pressure was applied for one minute. A thin layer of light cured resin was placed on the specimen surface. The specimens still retained in the mould were then placed in a thermostatically controlled oven at 37°C for 60 min. On removal from the moulds the specimens were checked for voids and chipped edges under a magnifying lens at x8 magnification. They were then stored in deionised distilled water in the oven for 23 hours before testing. The apparatus used for the shell test consists of an indentor and knife edge support which are mounted on a universal load testing machine (Hounsfield). The thickness of the specimen was measured three times across three different diameters as shown in Fig 4, the mean value being taken from them. The specimens were then loaded to failure, using a cross head speed of 0.5 mm/min and a 200 N load cell. The load at failure was recorded graphically on the chart record.
The bi-axial flexural strength was calculated using the formula:

\[ X = \frac{P}{h^2 \{ 0.606 \times \log_e \left( \frac{a}{h} \right) + 1.13 \} } \]

Where, \( X \) = Bi-axial flexural strength.

- \( P \) = load at failure.
- \( h \) = specimens thickness.
- \( a \) = the radius of support knife edge (4 mm) (Piddock et al 1984).

**Pilot Study 2:** (Specimens packed between metal plates with glaze applied and conditioned for 10 min).

The technique was as described in the first pilot study. However, metal plates were used instead of glass plates covered with a sheet of polyethylene placed one on
either side of the specimen. The two metal plates were clamped, and the assembly was placed in the oven for 10 min. The specimens were then removed from the moulds, a protective layer of resin applied over the whole specimen surface and cured for 20 seconds. Ten specimens were constructed for this study. The specimens were then stored in deionised distilled water for 23 hours 50 min. Testing was carried out at 24 hours from the time of the start of mixing.

**Pilot Study 3:** (Specimen packed between metal plates with glaze applied and conditioned for 60 min).

The technique was as described in pilot study two, except the conditioning period of the specimens in the mould was increased from 10 min to 60 min. The moulds and the plates were coated with a silicone spray instead of covering with a polyethylene sheet. The light cured resin was applied after removing the specimens from the oven. Ten specimens were constructed and then stored in deionised distilled water at 37 °C for 23 hours. Testing was carried out 24 hours after mixing.

**Pilot Study 4:** (Specimen packed between metal plates with no glaze applied and conditioned for 60 min).

The technique was as described in pilot study 3, except no resin was applied on the specimen surfaces. This was comparable with the pilot study 3 carried out for compression.

**Pilot Study 5:** (Change in maturation).

In this pilot study the technique for specimen manufacture was similar to pilot
study 4. The number of specimens was reduced to seven. These were stored in deionised distilled water for a week before testing at 37 °C. This was devised to determine the influence of maturation on mechanical properties of these materials.

Summary:

From the pilot studies, it was determined that all specimens would be mould stored for 60 minutes at 37 °C prior to removal. These were then stored in deionised distilled water at 37 °C until the appropriate test period had elapsed. No protective varnish was applied.
3.4 Main studies methods:

3.4.1 Compressive strength:

The materials investigated were the experimental high powder/liquid ratio (EXPT), conventional GIC, the alloy reinforced GIC (Hi-Dense) and the dental amalgam (Dispersalloy).

Cylindrical specimens (6 × 4 mm) were prepared using stainless steel split moulds. The internal surface of each mould and the metal plates were coated with a silicone spray (Releasil) to facilitate the removal of the hardened cement specimen.

All the materials were mixed according to the manufacturer’s instructions and packed to excess in the split mould. In order to consolidate the cement and avoid air entrapment the largest convenient portions of mixed cement were placed in the mould and placed to one side. The mould was packed to excess incrementally in this manner and then firmly seated on a metal plate. The extruded cement was removed, and a further metal plate placed in position on the top of the mould. The assembly was clamped together and then placed in a thermostatically controlled oven at 37°C within 3 minutes of the start of mixing and stored under these condition until 60 minutes after mixing had commenced to permit the cement to mature. Each specimen was then removed from the mould and checked for air-voids or chipped edges, using a magnifying lens at x8 magnification. Any exhibiting such defects were discarded. In order to prepare the large number of specimens required, these were produced in batches. Twelve specimens of each glass ionomer material were constructed in each batch. Specimens were then selected at random from each batch for storage for each time period. A total of six groups of ten specimens of each glass ionomer material were stored in deionised distilled water and a further six groups of ten of Hi-Dense alone were stored in artificial saliva at 37°C, as
described previously.

Three groups of ten specimens of dental amalgam were also constructed and randomly selected for storage for each time period and stored in deionised distilled water. Testing of the glass ionomer specimens was carried out at one day, one week, one month, three months, six months and one year after specimens preparation. Testing of the dental amalgam specimens was carried out at one day, one month and three months after specimens preparation.

Each specimen was placed between the plates of Hounsfield testing machine prior to loading at a cross head-speed 1 mm/min. The load was applied to the flat ends of the specimen. The load at failure was recorded on a strip-chart recorder. The compressive strength was calculated as described previously in compressive strength pilot study 1.

3.4.2 Uni-axial (Four point) flexural test (Soderholm 1986):

Both the reinforced and conventional (experimental, EXPT) GICs were investigated here together with the amalgam comparator.

Beam shaped specimens, 22 mm long × 2 mm width x 2 mm height, were prepared by packing the freshly prepared cement paste to excess into a stainless steel split mould coated on the internal surface with a silicone spray (Releasil). One capsule was used for each specimen. A metal plate coated with a silicone spray was placed over the exposed surface, the assembly was clamped and then allowed to set in a thermostatically controlled oven at 37°C for 60 minutes. The specimens were removed from the mould and checked for air-voids or chipped edges using a magnifying lens at x8 magnification. Any samples showing visible defects were discarded. Twelve specimens of each glass ionomer material were constructed in each batch. Specimens were selected at random.
from each batch for storage for each time period. Six groups of ten specimens of each
glass ionomer material were stored in deionised distilled water and a further six groups
of ten of Hi-Dense alone were stored in artificial saliva at 37°C as described previously.
Three groups of ten dental amalgam specimens were constructed and also randomly
selected for storage for each time period, and stored in deionised distilled water. These
acted as comparators. Testing of the glass ionomer specimens was carried out at one day,
one week, one month, three months, six months and one year after specimen preparation.
Testing of the dental amalgam specimens was carried out at one day, one month and
three months after specimen preparation. During testing the specimens were loaded to
failure in a four point bend testing jig using a Hounsfield, universal load testing machine,
at a cross head speed of 0.5 mm/min. The applied load was continuously registered on
a strip-chart recorder until failure occurs. The flexural strength was determined from the
formula:

\[ FS = \frac{6PA}{w \times h^2} \]

Where FS = flexure strength.

P = load at failure.

A = distance from lower span support to the closest upper load line.

w = width.

h = height.

3.4.3 Bi-axial, Ball-on-ring, flexural strength (shell test) (Wilshaw 1968):

The two materials evaluated here were a conventional GIC, experimental high
powder/liquid ratio, (EXPT) and alloy reinforced GIC, (Hi-Dense).

The materials were mixed according to manufacturer instruction. One capsule was used
for each specimen. A stainless steel mould, (10 mm in diameter, 1 mm thickness), was coated with a silicone spray and placed on a metal plate also coated with silicone spray. The cement was packed in the mould and another metal plate coated with silicone spray was placed over the exposed surface of the material. The assembly was clamped and then placed in a thermostatically controlled oven at 37°C for an hour to permit the cement to mature. On removal of the mould, the specimens were checked for air-voids or chipped edges under a magnifying lens at x8 magnification. Any sample showing visible defects was discarded. Six groups of ten specimens of both materials were constructed, and randomly selected for storage for each time period as described previously in the uni-axial test, and stored in deionised distilled water. A further six groups of ten of Hi-Dense alone were stored in artificial saliva at 37°C, as described previously.

Specimens were stored for each of the following time intervals prior to testing; one day, one week, one month, three months, six months and one year. The bi-axial flexural strength test was carried out using a universal load testing machine (Hounsfield). The specimens were placed on an annular knife edge with a diameter of 8 mm and then loaded to failure at a cross head speed of 0.5 min/mm and the load at failure recorded on a strip-chart recorder. The bi-axial flexural strength was calculated using the formula as described previously in shell pilot study 1 (Piddock et al 1984).

3.4.4 Evaluation of bond strength to enamel and dentine:

Again the materials investigated were the experimental high powder/liquid ratio, (EXPT), conventional GIC and Hi-Dense, the alloy reinforced GIC.

Two hundred freshly extracted, non-carious, human molar teeth were cleaned with an ultrasonic scaler and immersed in 10% formol saline for 10 minutes. They were then
washed with tap water and stored in deionised distilled water at 4°C until required for use. The teeth were divided into ten groups of ten, five were prepared for dentine bonding and the other five for enamel bonding. Teeth prepared for dentine bonding were sectioned horizontally at the maximum bulbosity of the crown with a rotary diamond saw to remove the occlusal portion of the clinical crown. The roots of the extracted teeth were sectioned. The extracted teeth were positioned with the occlusal surface (dentine bonding), and buccal surface (enamel bonding), facing downward in 11 mm (inside diameter) × 15 mm (depth) plastic ring moulds. The moulds were then filled with acrylic resin (De-Trey) to form the base. The preparation sequence is shown in Fig 5. Once the resin had set, the exposed dentine surfaces were sequentially abraded using 500, 800, and 1000 grit silicone carbide paper and water. All the specimens were kept in deionised distilled water at 37°C until the bonding of the cement was carried out.

The surface of each tooth was washed with deionised distilled water for 10 seconds and air dried for 5 seconds. Each surface was then conditioned using 25% polyacrylic for 30 seconds, rinsed with deionised distilled water for 10 seconds and dried with oil free air for 5 seconds. Both glass ionomer materials were mixed in a mechanical vibrator for 15 seconds as recommended by the manufacturer. One capsule was used for each specimen.
The mixed cement was condensed into a brass cylinder [3.20 mm (inside diameter) x 4 mm (height)], against an area of tooth surface. The excess cement around the brass cylinder at cement/tooth interface was removed using dental probe. The material was allowed to set at 37°C for one hour then stored in deionised distilled water for one day, one month, three months, six months and one year, until testing. Ten specimens were constructed for each test interval for each material for both enamel and dentine.

The specimens were inserted into a customised stainless steel jig and tested, as shown in Fig 6, at a cross head speed of 0.5 mm/min in universal load testing machine (Hounsfield H25K). The load required to produce bond failure was recorded on the strip chart and the type of bond failure for each specimen was checked using a magnifying lens at x8 magnification (Kerby 1992).
The force at failure was calculated using the formula:

$$ S = \frac{4 \, F}{\pi \, d^2} $$

Where $S =$ the force at failure in MPa, $F =$ Displacement load, $d =$ diameter of the specimen. (Coury et al 1982).

Representative specimens of the dentine surface before and after bonding were sputter coated with gold and observed using Scanning Electron Microscopy (SEM) at an accelerating voltage of 15 K V. These are shown in Fig 16, 17, 18 and 19.

3.4.5 **Erosion resistance: (Acid resistance).**

The materials evaluated for this part of the study were the experimental high powder/liquid ratio glass ionomer cement, and its metal reinforced counterpart.
Eighteen specimens of each material were constructed. The materials were mixed as described previously and packed into 4 mm diameter holes in stainless steel moulds. The materials were packed to excess and each mould was covered with two metal plates and clamped firmly. A one capsule mix was used for each specimen. The clamped moulds were conditioned at 37 °C for one hour to permit the cement to mature. Immediately after the conditioning period the specimens were blot dried and weighed (W1). The thickness (D1) of the specimen was measured using a micrometer screw gauge. The mean of five readings at different points on the specimen surface was taken. The specimens for each material were then divided into three groups of six. The first group was tested immediately after the one hour conditioning period. The second group was transferred into a bottle containing damp filter paper sealed securely with an airtight lid and placed in the humidity cabinet maintained at 37 ±1°C for 23 hours before testing, according to the standard method (Wilson et al 1986). The third group was stored in deionised distilled water for six months at 37 °C prior to testing.

Each specimen was subjected to a jet of 0.02 M lactic acid solution (pH 2.7) at 37 ±1°C, at a constant pressure, impinging on the surface of the cement at a constant flow rate of 120 ±4 ml/min. The flow rate was measured by running the device and sampling the fluid from every other jet (three jets) for one minute. The amount of the fluid collected was then measured. The specimens were placed in position in the test apparatus using a spacer to ensure that the centre of each specimen lay 10 mm vertically below the jet at the start of the experiment. The experiment was run for a maximum of 24 hours to avoid development of turbulent flow. The moulds were then removed, blot dried and all the specimens reweighed (W2) and the thickness remeasured (D2). The rate of erosion was calculated both by weight loss [weight loss per hour, (mg/h)] and height loss [loss
of specimen thickness per hour, (μm/h)] (Wilson et al 1986).

The rate of erosion (mg/h) was calculated from the equation:-

\[
R = \frac{(W_1 - W_2)}{T}
\]

Where  
\( R \) = rate of erosion.  
\( W_1 \) = weight before testing.  
\( W_2 \) = weight after testing.  
\( T \) = time.

The rate of erosion (μm/h) was calculated from the equation:-

\[
R = \frac{(D_1 - D_2)}{T}
\]

Where  
\( D_1 \) = thickness before testing.  
\( D_2 \) = thickness after testing.  
\( T \) = time.

3.4.6 Microleakage:

In this part of the study both glass ionomer materials were investigated.

Eighty freshly extracted, non-carious molar teeth were cleaned with an ultrasonic scaler and disinfected by soaking in 10% formol saline for 10 minutes. They were then stored in deionised distilled water at 4°C until required for use. Each tooth was mounted in an acrylic base (De-Trey). A class II preparation was made on each tooth, the design of the preparation, as shown in Fig 7, being as follows:-

- Preparations were either on mesial or distal side aspect of the tooth.
- The buccal-lingual width was at least one third the intercuspal dimension occlusally and one third the facial-lingual dimension interproximally.
- The depth of the occlusal portion was approximately 1 mm from the dentine-enamel junction into dentine.

- The proximal box extended approximately 1 mm below the cementum-enamel junction.

- The gingivo-axial width of the proximal box was approximately 1 mm from the gingival tooth surface.

The preparations were cleaned using pumice and a prophy brush in a slow handpiece to remove dentine chips and debris. They were then washed with deionised distilled water and the 25% solution of polyacrylic acid was applied for 30 seconds.

The preparations were washed with water for 15 seconds and dried with oil free compressed air for 5 seconds. An automatrix band coated with a silicone spray (Releasil) as a separating medium was placed around the tooth. The capsule was activated by the
application of pressure to the capsule guard for 3 seconds and mixed for 15 seconds in Silamat amalgamator (Vivadent). The mixed material was removed from the capsule using the spatula provided and condensed into the cavity using instruments moistened with a solution of isopropyl alcohol. The restorations were covered with light cured protected resin (Hi-glaze resin), provided by the manufacturer, and cured for 20 seconds. The restored teeth were then placed in a thermostatically controlled oven at 37°C for 60 minutes prior to removal of the band. Gross excess was removed with a 12 fluted carbide bur (Jet FG 7902) using a high speed handpiece. The teeth were divided into four groups of ten for each material and placed in deionised distilled water prior to load cycling. One unloaded group for each material acted as a control (n=10). The teeth from the other three groups for each material were load cycled immediately after the conditioning time for three different periods equivalent to one day, one week and one month. Each tooth was placed under a ball bearing, attached to the upper crosshead of a universal load testing machine (Instron, High Wycombe, UK). The ball bearing (12.25 mm diameter) was brought into contact with both buccal and lingual cusps of the tooth avoiding direct contact with the restoration. This permits an outward deflection of the cusps as the load was applied. The tooth was immersed in water during this time. The cycling time was one cycle per second. The cross head speed was 25 mm/min. The load applied was between 9 to 180 N, as utilised in a number of other studies (Anderson 1956, Anderson & Picton 1958, Helkimo & Ingerwall 1978, DeBoever 1978 & Gibbs 1981). Table 3 shows the number of cycles for each testing period. These were derived from the work of Delong and Douglas (1991) in which they assumed that 250,000 cycles in the artificial mouth equals one year in the human mouth.
Table 3: The cycling test.

<table>
<thead>
<tr>
<th>Duration</th>
<th>Number of cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>One day</td>
<td>685</td>
</tr>
<tr>
<td>One week</td>
<td>4808</td>
</tr>
<tr>
<td>One month</td>
<td>20833</td>
</tr>
</tbody>
</table>

Dye penetration:

The teeth were dried by blotting with tissue paper and compressed air taking care to avoid desiccation. Three layer of nail varnish were applied to the entire external surface of the tooth except for an area 1 mm peripheral to the cavity margin and the restoration. Each layer of nail varnish was allowed to dry before the next layer was applied. Neutral red dye solution was used to provide a contrasting colour in the areas where microleakage occurred. Neutral red dye could be easily identified against the restoration, and its use has been described by many authors, (Shortall 1982, Taylor & Lynch 1992 & Alani & Toh 1997). The teeth were immersed in 0.1% neutral red dye solution for 24 hours at 37°C. At the end of the 24 hour period, the teeth were removed from the solution and washed with water and scrubbed with a brush to remove traces of dye. They were then stored in deionised distilled water at room temperature prior to sectioning.

Sectioning:

The teeth were mounted on cylinder holders using sticky wax, and sectioned using
a 0.2 mm diamond band saw (Exact, Mederex, Germany). Teeth were sectioned once in a mesio-distal plane and four times in bucco-lingual plane, as shown in Fig 8. Each section was approximately 1 mm in thickness. The sections were stored in deionised distilled water at room temperature until required for scoring. Two section scores were noted for cervical and sixteen for occlusal dye penetration.

**Evaluation of sections:**

The sections were viewed under a Wild Photo Microscope at x15 magnification (Wild Heerbrugg Limited). Any dye penetration at the occlusal and cervical tooth-restoration interface was evaluated using the scoring system as shown in Fig 9.
Figure 9: Schematic diagram to show microleakage scoring system.

Scoring (S) at the cervical (C)/occlusal (O) tooth-restoration interface:

0. No dye penetration

½. Dye penetration less than ¼ the length of the cervical floor (C) or occlusal wall (O).

1. Dye penetration less than half the length of the cervical floor (C) or occlusal wall (O).

2. Dye penetration up to the full length of the cervical floor (C) or occlusal wall (O).

3. Dye penetration up to half the axial wall (C) or the occlusal floor.

4. Dye penetration up to the whole axial wall (C) or the occlusal floor (O).
3.5 Indirect assessment of occlusal wear:

3.5.1 Method:

One operator carried out this trial for assessment of occlusal wear in a general dental practice. Ethical approval was sought and gained from Dartford and Gravesham NHS Trust. All patients were required to provide informed written consent after being thoroughly briefed on all aspects of the study.

The criteria for prescribing restorative treatment were that each patient required the treatment of occlusal caries in two molar teeth within the same arch. Seventy restorations were placed in 35 patients, whose age ranged from 11 to 37 years. Two materials were used, a disperse phase alloy amalgam (Dispersalloy) and a metal reinforced glass ionomer cement (Hi-Dense). One restoration using each material was placed in each patient. Putty wash impressions were made using polyvinyl siloxane impression material [Extrude (Kerr) and Polytrays (Dentsply)]. These formed a record of the teeth and restorations. The impressions were taken at baseline (one week after insertion), 6 months and 12 months. The impressions were poured with dental stone (Crystacal D), and the restorations were presented as stone casts for an indirect assessment of materials wear at the three time periods.

3.5.2 Restoration procedures:

The operator placed all the restorations under local anaesthesia [Lignospan Special (Septodont)].

Conventional class I cavity preparations were determined by the amount of caries present. They were cut at high speed using pear-shaped diamond burs (Unodent 565,566), caries extending into dentine was removed with round steel burs (Unodent, 014,018) at slow
speed. Each cavity was washed, dried and lined with calcium hydroxide (Dycal). Every patient had two restorations inserted in accordance with the manufacturer’s instructions. The cavities for Hi-Dense restorations were conditioned for 10 seconds using 25\% polyacrylic acid (Shofu Hi-Tooth cleanser). They were then washed and air dried using a 3:1 syringe. The Hi-Dense capsules were mixed for 15 seconds in a mechanical mixer (Degussa Dentomat 2) using a capsule extension adaptor to ensure the correct mixing speed. The material was then condensed into the cavity and carved using a Hollenbach carver. It was then covered with a thin layer of light cured resin and this layer was cured for 10 seconds using a Luxor light (I.C.I).

The Dispersalloy capsules were mixed in mechanical mixer for 15 seconds. The amalgam was condensed into the cavities, carved and burnished. The occlusion was checked for both restorations. Both restorations were polished after one week using Shofu Greenie and Brownie. Petroleum jelly was applied to Hi-Dense restoration surface to minimise frictional heating and reduce desiccation. Finally light cured resin was re-applied on Hi-Dense surface and cured for 10 seconds. The occlusion of both restoration were checked again. The impressions were then taken for the baseline using polyvinyl siloxane (Extrude), and poured with dental stone (Crystacal D). The restorations were presented as stone casts for the base line. Other impressions were taken after 6 and 12 months and also presented as stone casts for an indirect assessment of materials wear.

3.5.3 Method of assessment:

The indirect assessment of occlusal wear was used in the evaluation of stone replicas. The restorations were assessed and independently scored by two examiners at baseline, 6 and 12 months using the Ivoclar-Vivadent system as shown in Fig 10.
Figure 10: Photograph of the Vivadent Ivoclar standard for cast evaluation.
This consists of tooth-sized replicas with restoration-like incremental defects ranging from 25-1000 μm. The scales were intended to be read in 25 μm increments from 0 to 200 μm, in 50 μm increments between 200 and 500 μm and 100 μm steps from 500 to 1000 μm (1.00 mm) (Cvar & Ryge 1971 & Taylor et al 1989). The two examiners had previously been trained to assess any defects ranging from 25 to 1000 μm and to determine the point where the greatest wear had occurred at the margin and then estimate the loss of material.

The training was achieved by asking each examiner to score the same number of stone replicas independently. The examiners then compared their scores and any obvious discrepancies were discussed and agreed a figure of wear. This was repeated until the examiners were in good agreement.

Each examiner split the restoration into three areas, buccal, lingual and the point where they considered that the greatest loss of material had occurred (highest point). Each examiner scored each area of each restoration independently. The two examiners’ scores were compared and any discrepancies greater than 50 μm were noted. The two examiners then conferred and reassessed these measurements.

3.5.4 Method of analysis:

Mean value for the two scores was taken. The loss of material for each restoration at each period of time was calculated by substraction of the baseline score from that of six and twelve months scores. Upper and lower arch restorations for each material were scored separately.
Chapter (4)

Results
4 Results:

All results were analysed using the Mann-Whitney test with confidence limit set at 95% level, using a Minitab program (release no. 6). The results will be presented with both mean and median. Since there were a limited number of specimens evaluated for each test at each test period, it was decided to evaluate the results using non-parametric statistics since it was not possible to rely on the results having a normal distribution.

4.1 Pilot studies results:

4.1.1 Compressive strength:

Pilot study 1: (Specimen with glaze applied and conditioned for 10 min).

The specimen dimensions, load at failure and compressive strength of the six specimens together with the mean value and standard deviation (sd.) are presented in Table 4. The mean strength of all specimens was 129.5 MPa (19.2). In this study the scatter of results shows a variation of 14%.

Table 4: Compressive strength: pilot study 1.

<table>
<thead>
<tr>
<th>Sam No.</th>
<th>Sample Diameter (mm).</th>
<th>Load at Failure</th>
<th>Compressive Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>D1</td>
<td>D2</td>
<td>D3</td>
</tr>
<tr>
<td>1</td>
<td>4.01</td>
<td>4.01</td>
<td>4.01</td>
</tr>
<tr>
<td>2</td>
<td>4.02</td>
<td>4.03</td>
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<td>3</td>
<td>4.03</td>
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<td>4.03</td>
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<tr>
<td>5</td>
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<td>4.00</td>
<td>4.03</td>
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<tr>
<td>6</td>
<td>4.01</td>
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</tr>
<tr>
<td>Mean</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Median</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

128
Pilot study 2: (Specimen with glaze applied and conditioned for 60 min):

The specimen dimensions, load at failure and compressive strength of eight specimens together with the mean value and standard deviation (sd.) are presented in Table 5. The mean strength of all specimens was 117.1 MPa (34.4). This was not significantly different from that noted in pilot study 1, (p ≤ 0.33). A wide scatter of results was noted with this study however with a variation of 29.3%. Two specimens were substantially below the strengths of all other samples. If these are excluded, the mean value is 132 MPa (24.5) which is very similar to that noted in pilot study 1. No obvious flaws were observed in the two specimens either before or after loading.

Table 5: Compressive strength: pilot study 2.

<table>
<thead>
<tr>
<th>Sam No.</th>
<th>Sample Diameter (mm)</th>
<th>Load at Failure</th>
<th>Compressive Strength</th>
</tr>
</thead>
<tbody>
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<td></td>
<td>D1</td>
<td>D2</td>
<td>D3</td>
</tr>
<tr>
<td>1</td>
<td>4.00</td>
<td>3.99</td>
<td>3.96</td>
</tr>
<tr>
<td>2</td>
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</tbody>
</table>

Pilot study 3: (Specimens which were surface ground with no glaze applied and conditioned for 60 min). Table 6 shows the specimen dimensions, load at failure and
compressive strength of fifteen specimens together with the mean value and standard deviation (sd.). The mean strength of all specimens was 201.6 MPa (16.7). This was statistically highly significantly greater than the strengths noted in both previous pilot studies (p ≤ 0.0005) and (p ≤ 0.0002). The scatter of results here was also less than that of the previous studies with a variation of 8.2%.

Table 6: Compressive strength: pilot study 3.

<table>
<thead>
<tr>
<th>Sam No.</th>
<th>Sample Diameter (mm)</th>
<th>Load at Failure</th>
<th>Compressive Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>D1</td>
<td>D2</td>
<td>D3</td>
</tr>
<tr>
<td>1</td>
<td>4.02</td>
<td>4.00</td>
<td>3.99</td>
</tr>
<tr>
<td>2</td>
<td>4.00</td>
<td>3.98</td>
<td>3.97</td>
</tr>
<tr>
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</tr>
<tr>
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<td>3.96</td>
<td>4.05</td>
</tr>
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<td>4.01</td>
<td>4.03</td>
<td>4.01</td>
</tr>
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<td>4.02</td>
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</tr>
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<td>4.02</td>
<td>3.99</td>
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<td>3.99</td>
<td>3.98</td>
<td>3.97</td>
</tr>
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<td>11</td>
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<td>3.98</td>
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<tr>
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<td>4.00</td>
<td>3.99</td>
</tr>
<tr>
<td>Median</td>
<td>4.00</td>
<td>4.00</td>
<td>3.99</td>
</tr>
</tbody>
</table>

Mean 201.6 MPa (sd. 16.7)
Median 198.6 MPa
Pilot study 4: (Specimens which were surface ground with glaze applied and conditioned for 60 min).

The specimen dimensions, load at failure and compressive strength of nine specimens together with the mean value and standard deviation (sd.) are presented in Table 7. The mean compressive strength was 179.1 MPa (33.1). This was again statistically significantly greater than the strengths noted in the two initial pilot studies \((p \leq 0.01)\) and \((p \leq 0.003)\). However, it was not statistically significantly different from the strengths obtained in pilot study 3, although the scatter of these results was substantially higher with a variation of 18.5%.

Table 7: Compressive strength: pilot study 4.

<table>
<thead>
<tr>
<th>Sam No.</th>
<th>Sample Diameter (mm)</th>
<th>Load at Failure</th>
<th>Compressive Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>D1</td>
<td>D2</td>
<td>D3</td>
</tr>
<tr>
<td>1</td>
<td>3.97</td>
<td>3.98</td>
<td>3.97</td>
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<td>3.97</td>
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<tr>
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<td>3.98</td>
<td>3.96</td>
<td>4.00</td>
</tr>
<tr>
<td>4</td>
<td>3.98</td>
<td>3.99</td>
<td>4.04</td>
</tr>
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<td>3.97</td>
<td>3.96</td>
<td>3.98</td>
</tr>
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<td>3.98</td>
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<td>3.96</td>
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<td>3.96</td>
<td>3.97</td>
</tr>
<tr>
<td>Median</td>
<td>3.96</td>
<td>3.96</td>
<td>3.97</td>
</tr>
</tbody>
</table>

131
4.1.2 Bi-axial flexural strength (Shell):

Pilot study 1:

(Specimens packed between glass slides with glaze applied and conditioned for 60 min).

The specimen dimensions, load at failure and flexural strength of six specimens together with the mean and standard deviation are presented in Table 8. The mean strength of all specimens was 33.4 MPa (8.9). The scatter of these results was very high with a variation of 26.6%.

Table 8: Bi-axial flexural strength: pilot study 1.

<table>
<thead>
<tr>
<th>Sam No.</th>
<th>Sample Thickness (mm)</th>
<th>Load at Failure</th>
<th>Shell Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T1 1.51</td>
<td>34.0 N</td>
<td>21.6 MPa</td>
</tr>
<tr>
<td></td>
<td>T2 1.48</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>T3 1.47</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Mean 1.48</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1.21</td>
<td>40.0 N</td>
<td>46.4 MPa</td>
</tr>
<tr>
<td></td>
<td>1.27</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.26</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Mean 1.24</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>1.44</td>
<td>39.0 N</td>
<td>38.6 MPa</td>
</tr>
<tr>
<td></td>
<td>1.40</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.46</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Mean 1.43</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>1.45</td>
<td>26.4 N</td>
<td>25.8 MPa</td>
</tr>
<tr>
<td></td>
<td>1.43</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.41</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>Mean 1.43</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>1.41</td>
<td>46.0 N</td>
<td>32.1 MPa</td>
</tr>
<tr>
<td></td>
<td>1.43</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.44</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Mean 1.42</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>1.53</td>
<td>59.8 N</td>
<td>35.9 MPa</td>
</tr>
<tr>
<td></td>
<td>1.47</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.54</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Mean 1.51</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Mean 33.4 MPa (sd. 8.9)
Median 34.0 MPa

Pilot study 2:

(Specimen packed between metal plates with glaze applied and conditioned for 10 min).

The specimen dimensions, load at failure and flexural strength of ten specimens together with the mean and standard deviation are presented in Table 9. The mean strength of all specimens was 29.6 MPa (4.8). There was no statistically significantly different between
the results of this study and these of the pilot study 1, \( p \leq 0.35 \). However, the scatter in results of this study was less than in pilot study 1 with a variation of 16.2%.

Table 9: Bi-axial flexural strength: pilot study 2.

<table>
<thead>
<tr>
<th>Sam No.</th>
<th>Sample Thickness (mm)</th>
<th>Load at Failure</th>
<th>Shell Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T1</td>
<td>T2</td>
<td>T3</td>
</tr>
<tr>
<td>1</td>
<td>1.18</td>
<td>1.12</td>
<td>1.23</td>
</tr>
<tr>
<td>2</td>
<td>1.39</td>
<td>1.25</td>
<td>1.24</td>
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<tr>
<td>3</td>
<td>1.22</td>
<td>1.28</td>
<td>1.31</td>
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<tr>
<td>4</td>
<td>1.20</td>
<td>1.23</td>
<td>1.16</td>
</tr>
<tr>
<td>5</td>
<td>1.18</td>
<td>1.15</td>
<td>1.16</td>
</tr>
<tr>
<td>6</td>
<td>1.30</td>
<td>1.38</td>
<td>1.39</td>
</tr>
<tr>
<td>7</td>
<td>1.10</td>
<td>1.12</td>
<td>1.13</td>
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<tr>
<td>8</td>
<td>1.34</td>
<td>1.19</td>
<td>1.15</td>
</tr>
<tr>
<td>9</td>
<td>1.19</td>
<td>1.26</td>
<td>1.24</td>
</tr>
<tr>
<td>10</td>
<td>1.15</td>
<td>1.24</td>
<td>1.18</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Median</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Pilot study 3:

(Specimen packed between metal plates with glaze applied and conditioned for 60 min).

Table 10 shows the specimen dimensions, load at failure and flexural strength of ten specimens together with the mean value and standard deviation. The mean strength of all specimens was 35.3 MPa (5.1). This was not statistically significantly different from that observed in pilot study 1, \( p \leq 0.7 \), but was significantly higher than in pilot study 2, \( p \leq 0.04 \). This current investigation showed less scatter in results with a variation of
14.4% when compared with pilot study one.

Table 10: Bi-axial flexural strength: pilot study 3.

<table>
<thead>
<tr>
<th>Sam No.</th>
<th>Sample Thickness (mm)</th>
<th>Load at Failure</th>
<th>Shell Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T1</td>
<td>T2</td>
<td>T3</td>
</tr>
<tr>
<td>1</td>
<td>1.16</td>
<td>1.07</td>
<td>1.12</td>
</tr>
<tr>
<td>2</td>
<td>1.15</td>
<td>1.14</td>
<td>1.20</td>
</tr>
<tr>
<td>3</td>
<td>1.16</td>
<td>1.21</td>
<td>1.17</td>
</tr>
<tr>
<td>4</td>
<td>1.17</td>
<td>1.20</td>
<td>1.21</td>
</tr>
<tr>
<td>5</td>
<td>1.18</td>
<td>1.22</td>
<td>1.22</td>
</tr>
<tr>
<td>6</td>
<td>1.13</td>
<td>1.16</td>
<td>1.12</td>
</tr>
<tr>
<td>7</td>
<td>1.12</td>
<td>1.20</td>
<td>1.20</td>
</tr>
<tr>
<td>8</td>
<td>1.20</td>
<td>1.16</td>
<td>1.19</td>
</tr>
<tr>
<td>9</td>
<td>1.13</td>
<td>1.15</td>
<td>1.17</td>
</tr>
<tr>
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<td>1.10</td>
<td>1.17</td>
<td>1.13</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Median</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Pilot study 4:

(Specimen packed between metal plates with no glaze applied and conditioned for 60 min). The specimen dimensions, load at failure and flexural strength of unprotected surfaces of ten specimens together with the mean value and standard deviation are presented in Table 11. The mean strength of all the specimens was 39.4 MPa (4.9). This strength was not statistically significantly different from the strengths noted for the pilot study 1, \( p \leq 0.1 \) and pilot study 3, \( p \leq 0.08 \). However, the strength was highly significantly greater than pilot study 2, \( p \leq 0.0006 \). The scatter in results was slightly
better than in pilot study 3 with a variation of only 12.4%.

Table 11: Bi-axial flexural strength: pilot study 4.

<table>
<thead>
<tr>
<th>Sam No.</th>
<th>Sample Thickness (mm)</th>
<th>Load at Failure</th>
<th>Shell Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T1</td>
<td>T2</td>
<td>T3</td>
</tr>
<tr>
<td>1</td>
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</tr>
<tr>
<td>4</td>
<td>1.15</td>
<td>1.13</td>
<td>1.12</td>
</tr>
<tr>
<td>5</td>
<td>1.15</td>
<td>1.15</td>
<td>1.17</td>
</tr>
<tr>
<td>6</td>
<td>1.19</td>
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<td>1.18</td>
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<tr>
<td>7</td>
<td>1.10</td>
<td>1.10</td>
<td>1.10</td>
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<td>1.15</td>
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<td>1.25</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Median</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Pilot study 5: (Change in maturation)

Table 12 shows the specimen dimensions, the load at failure at seven days and flexural strength of the specimens together with the mean value and standard deviation. The mean strength of all specimens at 7 days was 36.8 MPa (3.5). The strength was not statistically significantly different from the strength of pilot study 1, (p ≤ 0.5), study 3, (p ≤ 0.4) and study 4, (p ≤ 0.4). However, the strength was highly significantly greater than that observed for study 2, (p ≤ 0.007). The scatter in
The results of this study was better than all the studies with a variation of 9.5%.

Table 12: Bi-axial flexural strength: pilot study 5.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Sample Thickness (mm)</th>
<th>Load at Failure</th>
<th>Shell Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T1</td>
<td>T2</td>
<td>T3</td>
</tr>
<tr>
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</tr>
<tr>
<td>5</td>
<td>1.12</td>
<td>1.05</td>
<td>1.11</td>
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</tr>
<tr>
<td>7</td>
<td>1.14</td>
<td>1.20</td>
<td>1.25</td>
</tr>
</tbody>
</table>

Mean 36.8 MPa (sd. 3.5)
Median 37.8 MPa
4.2 Results:

4.2.1 Mechanical properties of comparative material (amalgam):

The mean values of compressive and flexural strengths and standard deviation (sd.) of the disperse phase dental amalgam (Dispersalloy), which acted as a comparator for these tests are presented in Table 13. This material showed high early compressive strength 387.6 MPa (29.4) within the first 24 hours and 138.8 MPa (18) for uni-axial flexural strength (four point). The material maintained its strength at six months, the compressive strength was 383.7 MPa (36.2) and the uni-axial flexural strength was 148.6 MPa (28.3).

Table 13: Compressive and flexural strengths of amalgam (MPa).

<table>
<thead>
<tr>
<th>Duration</th>
<th>Compressive Strength</th>
<th>St. Dev</th>
<th>Median</th>
<th>4 point Strength</th>
<th>St. Dev</th>
<th>Median</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Day</td>
<td>387.6</td>
<td>29.4</td>
<td>387.0</td>
<td>138.8</td>
<td>18.0</td>
<td>141.9</td>
</tr>
<tr>
<td>1 Month</td>
<td>333.9</td>
<td>27.6</td>
<td>331.6</td>
<td>149.7</td>
<td>19.4</td>
<td>143.4</td>
</tr>
<tr>
<td>6 Months</td>
<td>383.7</td>
<td>36.3</td>
<td>382.3</td>
<td>148.6</td>
<td>28.3</td>
<td>151.7</td>
</tr>
</tbody>
</table>

4.2.2 Compressive strength:

The mean values of compressive strength of both glass ionomer materials at six test periods together with standard deviation (sd.) are shown in Table 14 and Fig 11. The two materials showed high initial strength values at one day, 220.4 MPa (9.8) for Hi-Dense and 226.5 MPa (10.6) for experimental high powder/liquid (EXPT). The compressive strength of both materials increased with respect to time. The peak value for Hi-Dense was achieved at three months whereas that of the experimental was effective
Fig (11): Compressive strength of glass ionomer cement.
after one month. Both materials maintained their strengths thereafter. There was no significant difference between Hi-Dense and EXPT at all test periods apart from one month. At this time interval, EXPT was highly significant stronger, (p ≤ 0.005).

Table 14: Compressive strength of GICs (MPa).

<table>
<thead>
<tr>
<th>Duration</th>
<th>Hi-Dense</th>
<th>St. Dev</th>
<th>Median</th>
<th>EXPT</th>
<th>St. Dev</th>
<th>Median</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Day</td>
<td>220.4</td>
<td>9.8</td>
<td>219.9</td>
<td>226.5</td>
<td>10.6</td>
<td>226.2</td>
</tr>
<tr>
<td>1 Week</td>
<td>254.2</td>
<td>17.8</td>
<td>251.4</td>
<td>262.8</td>
<td>14.4</td>
<td>259.7</td>
</tr>
<tr>
<td>1 Month</td>
<td>254.4</td>
<td>20.4</td>
<td>250.4</td>
<td>283.1</td>
<td>15.8</td>
<td>281.0</td>
</tr>
<tr>
<td>3 Months</td>
<td>283.4</td>
<td>22.5</td>
<td>290.3</td>
<td>287.8</td>
<td>18.2</td>
<td>291.9</td>
</tr>
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<td>6 Months</td>
<td>284.7</td>
<td>21.1</td>
<td>279.3</td>
<td>275.5</td>
<td>25.6</td>
<td>270.3</td>
</tr>
<tr>
<td>1 Year</td>
<td>279.9</td>
<td>12.4</td>
<td>275.7</td>
<td>277.7</td>
<td>23.9</td>
<td>284.2</td>
</tr>
</tbody>
</table>

4.2.3 Uni-axial flexural strength (Four point):

Table 15 and Fig 12 show the mean values and standard deviations obtained for the uni-axial, four point flexural strength test of the two glass ionomer materials. The Hi-Dense strength at one day was 43.3 MPa (7.9) and EXPT strength was 40.8 MPa (7.7). The initial one day results showed that there was no significant difference between Hi-Dense and EXPT. However, at the later test times Hi-Dense was statistically significantly stronger than EXPT cement, [one week (p ≤ 0.01), one month (p ≤ 0.007), three months (p ≤ 0.011), six months (p ≤ 0.04), and one year (p ≤ 0.04)]. Overall, Hi-Dense cement showed a statistically significant increase in strength up to three months (p ≤ 0.002) and this increase was maintained for the later test intervals. The EXPT cement showed a slight increase in strength but there was no statistically significant difference between any of the values obtained at the various test interval.
Fig (12): Uni-axial (Four point) strength of glass ionomer cement.
Table 15: Uni-axial (4 point) flexural strength of GICs (MPa).

<table>
<thead>
<tr>
<th>Duration</th>
<th>Hi-Dense</th>
<th>St. Dev</th>
<th>Median</th>
<th>EXPT</th>
<th>St. Dev</th>
<th>Median</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Day</td>
<td>43.3</td>
<td>7.9</td>
<td>41.9</td>
<td>40.9</td>
<td>7.7</td>
<td>41.3</td>
</tr>
<tr>
<td>1 Week</td>
<td>51.8</td>
<td>9.8</td>
<td>49.7</td>
<td>39.5</td>
<td>7.5</td>
<td>39.2</td>
</tr>
<tr>
<td>1 Month</td>
<td>51.4</td>
<td>11.0</td>
<td>47.1</td>
<td>41.6</td>
<td>3.3</td>
<td>41.0</td>
</tr>
<tr>
<td>3 Months</td>
<td>57.9</td>
<td>8.0</td>
<td>57.2</td>
<td>46.8</td>
<td>8.0</td>
<td>45.9</td>
</tr>
<tr>
<td>6 Months</td>
<td>56.6</td>
<td>7.1</td>
<td>58.5</td>
<td>48.6</td>
<td>8.4</td>
<td>45.5</td>
</tr>
<tr>
<td>1 Year</td>
<td>55.2</td>
<td>13.2</td>
<td>51.4</td>
<td>43.2</td>
<td>11.1</td>
<td>40.9</td>
</tr>
</tbody>
</table>

4.2.4 Bi-axial strength, (Shell):

The results of bi-axial flexural strength are presented in Table 16 and Fig 13, showing the mean strength values and standard deviation. The Hi-Dense strength at one day was 51.4 MPa (5.1) and EXPT strength was 50.7 MPa (4.9). These results showed that Hi-Dense material increased and maintained its strength over a long period of time. The EXPT material could only maintain its strength for one month after which there was a significant reduction.

There was no significant difference between Hi-Dense and EXPT at the one day period or after one month storage period. However, Hi-Dense was significantly stronger at one week, three months and all later time intervals, [one week (p ≤ 0.002), three months (p ≤ 0.02), six months (p ≤ 0.0002) and year (p ≤ 0.002)]. At one month the considerable scatter of values for the Hi-Dense may have masked the general trend in strength values observed.
Fig (13): Bi-axial (Shell) strength of glass ionomer cement.
Table 16: Bi-axial (shell) flexural strength of GICs (MPa).

<table>
<thead>
<tr>
<th>Duration</th>
<th>Hi-Dense</th>
<th>St. Dev</th>
<th>Median</th>
<th>EXPT</th>
<th>St. Dev</th>
<th>Median</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Day</td>
<td>51.4</td>
<td>5.1</td>
<td>50.3</td>
<td>50.7</td>
<td>5.0</td>
<td>50.0</td>
</tr>
<tr>
<td>1 Week</td>
<td>56.4</td>
<td>4.5</td>
<td>55.6</td>
<td>48.8</td>
<td>3.6</td>
<td>47.4</td>
</tr>
<tr>
<td>1 Month</td>
<td>50.3</td>
<td>5.6</td>
<td>49.6</td>
<td>52.5</td>
<td>4.7</td>
<td>52.0</td>
</tr>
<tr>
<td>3 Months</td>
<td>55.0</td>
<td>6.3</td>
<td>56.1</td>
<td>47.8</td>
<td>4.2</td>
<td>48.1</td>
</tr>
<tr>
<td>6 Months</td>
<td>54.9</td>
<td>3.0</td>
<td>55.0</td>
<td>43.5</td>
<td>4.4</td>
<td>42.3</td>
</tr>
<tr>
<td>1 Year</td>
<td>53.7</td>
<td>6.1</td>
<td>55.7</td>
<td>45.5</td>
<td>2.6</td>
<td>46.3</td>
</tr>
</tbody>
</table>

The results of the materials stored in artificial saliva are included in Appendix 1, 2 & 3.

Considerable difficulty was found in maintaining the pH of the solution stable for any length of time. For this reason the results were not included in the study.

4.2.5 Bonding strength to dentine and enamel:

The means and standard deviation of the measured bond strengths to dentine are shown in Table 17 and Fig 14. The initial bond strength of Hi-Dense to dentine (one day) was 4.8 MPa (1.1) and the EXPT bond strength was 6.1 MPa (0.7). Statistically the bond strength of Hi-Dense was significantly lower than that of EXPT at this early stage, \( p \leq 0.011 \). The increase in bond strength of Hi-Dense between one day and one month was highly significant, \( p \leq 0.002 \) but this was reduced significantly by three months, \( p \leq 0.03 \). There was a very substantial scatter of results at the one month time interval. At six months the material showed a higher value, \( p \leq 0.01 \) which was maintained at later period.

The bond strength of the EXPT material was highly significantly greater at three months, \( p \leq 0.0007 \) and this was maintained at subsequent test periods.
Fig (14): Bond strength of glass ionomer cement to dentine.
At the end of the experiment, the bond strength of EXPT to dentine was significantly stronger, \((p \leq 0.04)\) than that of Hi-Dense.

The results of bond strength measurements to enamel are presented in Table 17 and Fig 15. At one day the bond strength of Hi-Dense to enamel was 11.7 MPa (2.7) and that of EXPT was 8.1 MPa (0.6). The results showed that the bond strength of Hi-Dense was highly significantly stronger than EXPT at one day, \((p \leq 0.003)\) and one month, \((p \leq 0.002)\). The bond strengths of both materials increased significantly at three months, [Hi-Dense, \((p \leq 0.007)\) and EXPT \((p \leq 0.0002)\)]. This increase was maintained at later tests interval. There was no significant difference between the materials at the later test intervals.

Table 17: Bond strength of GICs to dentine and enamel.

|                        | Dentine Bonding (MPa) |                                |                                |                                |                                |                                |                                |                                |
|------------------------|----------------------|--------------------------------|--------------------------------|--------------------------------|--------------------------------|--------------------------------|--------------------------------|                                |
|                        | Duration             | Hi-Dense | St. Dev. | Median | EXPT | St. Dev. | Median |                                |                                |                                |                                |
|                        | 1 Day                | 4.8      | 1.1      | 4.9    | 6.1  | 0.8      | 6.3    |                                |                                |                                |                                |
|                        | 1 Month               | 7.5      | 2.1      | 7.6    | 6.4  | 1.1      | 6.6    |                                |                                |                                |                                |
|                        | 3 Months              | 5.8      | 1.1      | 5.5    | 8.1  | 1.0      | 7.9    |                                |                                |                                |                                |
|                        | 6 Months              | 7.6      | 1.0      | 7.2    | 8.3  | 1.0      | 8.3    |                                |                                |                                |                                |
|                        | 1 Year                | 7.1      | 1.3      | 6.5    | 8.1  | 1.0      | 8.2    |                                |                                |                                |                                |
|                        | Enamel Bonding (MPa)  |                                |                                |                                |                                |                                |                                |                                |                                |                                |
|                        | 1 Day                | 11.7     | 2.7      | 11.6   | 8.1  | 0.6      | 8.2    |                                |                                |                                |                                |
|                        | 1 Month               | 11.3     | 1.8      | 11.2   | 9.1  | 0.5      | 9.1    |                                |                                |                                |                                |
|                        | 3 Months              | 13.7     | 0.8      | 13.4   | 12.8 | 1.9      | 12.9   |                                |                                |                                |                                |
|                        | 6 Months              | 12.4     | 1.6      | 12.4   | 12.5 | 1.4      | 12.3   |                                |                                |                                |                                |
|                        | 1 Year                | 13.7     | 2.3      | 12.9   | 12.3 | 0.8      | 12.2   |                                |                                |                                |                                |

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Figure 15: Bond strength of glass ionomer cements to enamel.
Representative specimens of the dentine surface before bonding to the Hi-dense were observed under Scanning Electron Microscopy (SEM) in two different magnifications (x50 & x200) (Field of view: 105 μm & 426 μm respectively), and are shown in Fig 16 & 18. The representative specimens after dentine surface bonding are shown in Fig 17 & 19 (Field of view: 103 μm & 432 μm).

Fig 16 and 18 show a flat dentine surfaces with remnants of the smear layer, clearly seen in low magnification (Field of view: 105 μm).

Fig 17 shows the material structure, (Hi-Dense), of different particle sizes and also remnant of the matrix distributed around these particles (Field of view: 103 μm).

Fig 19 shows particles embedded in the matrix (Field of view: 432 μm).

The material particles shown on the dentine surface suggested that the material failed cohesively to the dentine.

Mode of failure:

The type of bond failure to enamel and dentine was checked for each specimen in each period of time and presented as percentage in Table 18. The bond failure mode to enamel was mainly cohesive at all test period and few combined (adhesive and cohesive), but no pure adhesive failure has been noted. However, the bond failure mode to dentine was mainly combined failure (mostly adhesive) and a little adhesive but almost no pure cohesive failure has been noted.
Figure 16: Scanning electronic micrograph of dentine surface before bonding (Field of view: 105 μm)
Figure 17: Scanning electronic micrograph of dentine surface after bonding (Field of view: 103 μm).
Figure 18: Scanning electronic micrograph of dentine surface before bonding (Field of view: 426 µm).
Figure 19: Scanning electronic micrograph of dentine surface after bonding (Field of view: 432 μm).
Table 18: Type of GICs bond failure to enamel and dentine.

<table>
<thead>
<tr>
<th>Duration</th>
<th>Hi-Ds</th>
<th>EXPT</th>
<th>Hi-Ds</th>
<th>EXPT</th>
<th>Hi-Ds</th>
<th>EXPT</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Day</td>
<td>-</td>
<td>-</td>
<td>70</td>
<td>90</td>
<td>30</td>
<td>10</td>
</tr>
<tr>
<td>1 Month</td>
<td>-</td>
<td>-</td>
<td>80</td>
<td>80</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>3 Months</td>
<td>-</td>
<td>-</td>
<td>60</td>
<td>70</td>
<td>40</td>
<td>30</td>
</tr>
<tr>
<td>6 Months</td>
<td>-</td>
<td>-</td>
<td>70</td>
<td>80</td>
<td>30</td>
<td>20</td>
</tr>
<tr>
<td>1 Year</td>
<td>-</td>
<td>-</td>
<td>70</td>
<td>80</td>
<td>30</td>
<td>20</td>
</tr>
</tbody>
</table>

Mode of enamel bonding failure

Mode of dentine bonding failure

Combined mode of failure, * = mostly cohesive.

** = mostly adhesive.

4.2.6 Erosion resistance:

Table 19 and Fig 20 show the mean values and standard deviations of the erosion rate of glass ionomer materials measurements as height loss (μm/h). The 24 hours erosion rate of EXPT was 7.4 μm/h (2.3) and the Hi-Dense was 2.3 μm/h (0.3). The results showed that the erosion rate (μm/h) of the EXPT cement was highly significantly greater than Hi-Dense cement when tested at one hour, (p ≤ 0.005) and one day, (p ≤ 0.005) after mixing.
Fig (20): Erosion Rate (\( \mu \text{m/h} \)) of glass ionomer cement.

![Bar chart showing erosion rate over time for glass ionomer cement.](image-url)
However, the erosion rate of both cements were not significantly different after six months storage in deionised distilled water. The rates of erosion (µm/h) decreased as the storage period in deionised distilled water increased.

Table 19 and Fig 21 show the erosion rate of both materials as weight loss (mg/h). The 24 hours erosion rate of EXPT was 0.25 mg/h (0.03) and the Hi-Dense was 0.08 mg/h (0.02). The erosion rate, weight loss, (mg/h) of the EXPT was highly significantly greater than Hi-Dense at all testing periods (p \leq 0.005). The erosion rates for both materials were reduced when storage time in deionised distilled water increased.

Table 19: Erosion resistance of glass ionomer cements.

<table>
<thead>
<tr>
<th>Duration</th>
<th>Hi-Dense</th>
<th>sd.</th>
<th>Median</th>
<th>EXPT</th>
<th>sd.</th>
<th>Median</th>
</tr>
</thead>
<tbody>
<tr>
<td>One hour</td>
<td>2.9</td>
<td>0.3</td>
<td>2.9</td>
<td>8.7</td>
<td>1.0</td>
<td>8.9</td>
</tr>
<tr>
<td>One day</td>
<td>2.3</td>
<td>0.3</td>
<td>2.4</td>
<td>7.4</td>
<td>2.3</td>
<td>7.9</td>
</tr>
<tr>
<td>6 months</td>
<td>0.8</td>
<td>0.7</td>
<td>0.9</td>
<td>0.9</td>
<td>0.1</td>
<td>1.0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Erosion Rate (mg/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>One hour</td>
</tr>
<tr>
<td>One day</td>
</tr>
<tr>
<td>6 months</td>
</tr>
</tbody>
</table>
Fig (21): Erosion rate (mg/h) of glass-ionomer cement.
4.2.7 Microleakage:

The occlusal and cervical penetration (leakage) of dye around both glass ionomer materials are shown in Table 20. A total of 20 sections of each group cervically and 160 sections of each group occlusally were examined and scored. Most dye penetration was limited to a score of one at both margins for both materials, and little deeper penetration occurred at the occlusal margin. No scores of more than 2 were observed. Only four of the 640 sections examined for Hi-Dense and twelve of the 640 sections examined for EXPT achieved a score of 2. At the occlusal margins the EXPT material showed highly significantly more leakage only after one month cycling when compared to Hi-Dense, \( p \leq 0.002 \). There was no significant difference between the materials in terms of cycle penetration at either occlusal or cervical margins when the specimens remained unloaded. However, the results showed that at cervical margins, the EXPT material showed more dye penetration than the section scored for Hi-dense at all time periods \( \text{one day } p \leq 0.002 \text{ & one month } p \leq 0.03 \) when loading occurred. Mechanical loading with respect to time produced no major influence on leakage patterns at cervical margins of both materials and at occlusal margin of EXPT materials. However, there was less penetration of dye at occlusal margins on specimens of Hi-Dense after the equivalent of one month loading.
Table 20: GICs microleakage. (no. of section showing individual scoring)

<table>
<thead>
<tr>
<th>Cycling time</th>
<th>Microleakage of GIC (Hi-Dense).</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cervical penetration.</td>
<td>Occlusal penetration</td>
</tr>
<tr>
<td></td>
<td>S0</td>
<td>S½</td>
</tr>
<tr>
<td>Unloaded</td>
<td>14</td>
<td>6</td>
</tr>
<tr>
<td>One day</td>
<td>13</td>
<td>1</td>
</tr>
<tr>
<td>One week</td>
<td>8</td>
<td>6</td>
</tr>
<tr>
<td>One month</td>
<td>12</td>
<td>5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Cycling time</th>
<th>Microleakage of GIC (EXPT).</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cervical penetration</td>
<td>Occlusal penetration</td>
</tr>
<tr>
<td></td>
<td>S0</td>
<td>S½</td>
</tr>
<tr>
<td>Unloaded</td>
<td>7</td>
<td>7</td>
</tr>
<tr>
<td>One day</td>
<td>5</td>
<td>10</td>
</tr>
<tr>
<td>One week</td>
<td>6</td>
<td>5</td>
</tr>
<tr>
<td>One month</td>
<td>4</td>
<td>7</td>
</tr>
</tbody>
</table>

S = Score.
4.3 Indirect occlusal wear evaluation:

The amalgam and GIC wear results are shown in Table 21 and in bar chart form in Fig 22. The average amount of wear of the materials at six months was 23 μm for amalgam and 138 μm for the reinforced GIC (Hi-Dense). The average amount of wear of the materials at 12 months was 32 μm for amalgam and 225 μm for Hi-Dense. There was no significant difference between the amount of amalgam wear at 6 and 12 months (p ≤ 0.1). However, the amount of GIC wear at 12 months was highly significantly greater than at 6 months, (p ≤ 0.001). The tooth position (upper or lower) had no significant influence on the amount of wear for either materials (p ≤ 0.5). Overall, the amount of wear of GIC was highly significantly greater than amalgam at both time intervals evaluated.

Table 21: Teeth wear of stone models.

<table>
<thead>
<tr>
<th>Duration</th>
<th>Upper amalgam</th>
<th>Lower amalgam</th>
<th>Amalgam wear</th>
<th>Upper GIC</th>
<th>Lower GIC</th>
<th>GIC wear</th>
</tr>
</thead>
<tbody>
<tr>
<td>6 Months</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B W</td>
<td>19</td>
<td>21</td>
<td>23</td>
<td>114</td>
<td>159</td>
<td>138</td>
</tr>
<tr>
<td>L W</td>
<td>22</td>
<td>23</td>
<td>23</td>
<td>129</td>
<td>125</td>
<td></td>
</tr>
<tr>
<td>G W</td>
<td>27</td>
<td>27</td>
<td>27</td>
<td>142</td>
<td>159</td>
<td></td>
</tr>
<tr>
<td>12 Months</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B W</td>
<td>24</td>
<td>38</td>
<td>32</td>
<td>188</td>
<td>210</td>
<td>225</td>
</tr>
<tr>
<td>L W</td>
<td>26</td>
<td>32</td>
<td>32</td>
<td>204</td>
<td>246</td>
<td></td>
</tr>
<tr>
<td>G W</td>
<td>31</td>
<td>44</td>
<td>32</td>
<td>234</td>
<td>265</td>
<td></td>
</tr>
</tbody>
</table>

B W = Buccal wear.
L W = Lingual wear.
G W = Greater area of wear.
Fig (22): Tooth wear of stone models.
4.4 Elastic modulus:

The relationship between stress and strain is one of the important features of the mechanical behaviour of a material. The Young’s modulus of both glass ionomer materials was determined from the loading charts of the compressive strength test. The results are presented in Table 22.

This calculated by using the formula:-

\[ E = \sigma / e \]

where \( E \) = Young’s modulus,

\( \sigma \) = stress.

\( e \) = strain.

The results showed that there were no significant difference in elastic modulus between the two materials. The different test times had no effect on the elastic modulus of either material.

Table 22: Young’s modulus of glass ionomer cements (GPa).

<table>
<thead>
<tr>
<th>Duration</th>
<th>Hi-Dense</th>
<th>sd.</th>
<th>EXPT</th>
<th>sd.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Day</td>
<td>3.02</td>
<td>73.9</td>
<td>2.95</td>
<td>42.2</td>
</tr>
<tr>
<td>1 Week</td>
<td>3.02</td>
<td>78.4</td>
<td>2.98</td>
<td>66.5</td>
</tr>
<tr>
<td>1 Month</td>
<td>3.03</td>
<td>70.7</td>
<td>3.01</td>
<td>28.4</td>
</tr>
<tr>
<td>3 Months</td>
<td>3.03</td>
<td>74.9</td>
<td>3.02</td>
<td>90.4</td>
</tr>
<tr>
<td>6 Months</td>
<td>3.04</td>
<td>87.2</td>
<td>2.95</td>
<td>54.3</td>
</tr>
<tr>
<td>1 Year</td>
<td>3.04</td>
<td>58.4</td>
<td>3.00</td>
<td>47.8</td>
</tr>
</tbody>
</table>
Chapter (5)

Discussion
Discussion:

5.1 Materials:

Glass ionomer cements are considered by the dental profession as suitable for use in areas of erosion or Class III and V cavities. They have supposed cariostatic properties attributed to their capability to leach fluoride over a long period (Forsten 1977, Wilson et al 1985 & Mount 1995). They will also take-up and re-release fluoride (Williams et al 1997). An additional advantage is their adherence to both enamel and dentine (Aboush & Jenkins 1986, 1987). For these reasons the materials were considered to be worth of more general use in the oral cavity.

Despite these advantages, the early materials were brittle and had poor handling properties. In particular the setting reaction was extended. The material set clinically within a relatively short time, but the maturation phase was long. This means that the strength of the materials was inadequate for use in the posterior sextant of the mouth. Failure occurred due to wash out and occasionally fracture.

Attempts have been made to enhance the properties immediately after completion of mix. This was by use of copolymers of polyacrylic acid and increase in molecular weight of the acid. Considerable advances in the latter area were achieved by vacuum drying the acid. Even with these advances the materials were still relatively weak and brittle. The maturation phase was however still extended and the materials did not achieve their full mechanical properties until several weeks after placement (Williams & Billington 1989 & 1991 and Pearson & Atkinson 1991). It was also clear that it was frequently very difficult to achieve an acceptable mix for clinical use which fulfils the manufacturer's aim of a high powder liquid (P/L) ratio.

It was established that increasing the P/L ratio would increase the mechanical properties.
However, increasing the P/L ratio was practically difficult particularly when the mixing liquid was of high viscosity. Also there is a point after which it is not practical to increase the P/L as the material can not be mixed successfully by hand to a condensable mass. Encapsulation was introduced in part to improve this and in part to produce a consistent P/L ratio material.

The glass ionomer materials used in this study have a high P/L ratio. The experimental GIC (EXPT) has a P/L ratio of 4:1. The Hi-Dense has a similar composition to the EXPT with additional of an alloy, but its P/L ratio of glass to acid is slightly higher (approximately 4.38:1). These materials are both presented in an encapsulated form. This is likely to reduce the material porosity and ensure better consistency of mixing, which in turn ensures better condensation in both the specimen moulds and tooth cavities through the mechanical mixing. The P/L ratio of the EXPT material is considerably higher than many conventional available materials.

Although the two glass ionomer materials used in this study have the advantages described earlier that are attractive to the dentist, their main disadvantage is their handling properties because they are technique sensitive. In particular they are susceptible to moisture contamination or dehydration during the early stages of the setting reaction. Consequently the materials are placed in the oral cavity after mixing and then protected with a varnish or resin to reduce the risk of change in the water balance of the cement as it matures.

There are a number of contributory factors which influence the GIC’s performance clinically. These include:-
1) The difference in glass compositions and surface treatment which could influence the rate of setting of the GIC.

2) The handling of the material during mixing.

The glass composition can be varied, in particular the ratio of sodium to aluminium will affect the behaviour of glass and also affect the translucency. The level of acid washing which causes depletion of calcium and aluminium from the outer layer of glass will also be important. This may be used to vary the speed of set of the material. Variations in mixing are critical, in particular failure of the operator to use the correct powder/liquid ratio can seriously affect the performance of the materials (Billington et al 1990). To avoid the variations attributed to the operator, encapsulated materials were used and mechanically mixed. However, even encapsulated materials can show some limitations which can significantly affect the GIC performance, such as:-

- capsules which also permit injection of the mixed material have been shown to deliver paste at the incorrect P/L ratio. This will affect materials strength (Gee & Pearson 1993). Furthermore, it is mechanically essential that the P/L ratio be kept low enough to ensure that the paste can be injected from the capsule (Kilpatrick et al 1994).

- in materials where the polyacid is vacuum dried, water can not be placed in the pillow alone, because successful mixing will not occur. In this case a blend of a lower molecular weight acid and water is made. This means that encapsulated mechanically mix materials are slightly less strong than their hand mixed counterpart. This confirms the statement by Prosser et al (1986) that higher molecular weight polyacids are more difficult to get in solution within the capsules.

- the type of mechanical mixing machine used, in particular the amplitude of throw and the speed of the mixer can influence the properties of the mixed material (Gee & Pearson
- the manipulation of the capsule prior to mixing may lead to variation in performance of material. Failure to apply sufficient pressure to the pillow containing the liquid for sufficient time can also lead to considerable variations in P/L ratio and subsequent differences in material behaviour and performance.

These limitations and variations have been considered during the specimens preparation.

The addition of alloy to the GIC was initially to provide radiopacity, but the report of Kerby and Bleiholder (1991) suggested that an optimised addition of alloy enhanced mechanical properties. Early work appeared to confirm this (Coury et al 1986). The role of the alloy is not clear. It may just act as a material filler, but it has been shown that the oxide layer which forms on the surfaces of the alloy particles may react with the polyacid. This can form an effective chemical bond between the oxide films of metal particles and the carboxylate groups of polyacid which enhances the strength (Hotz et al 1977). Scanning electronic microscopy examination also revealed adhesion between the polyacrylate cement matrix and the surfaces of the particles (Kerby & Bleiholder 1991).

Little work has been undertaken to verify whether the optimum alloy addition really does increase mechanical properties, although Billington et al (1990) & Williams et al (1992a) did show that there was an increase in strength over non-addition counterpart. Increases in P/L ratio may have modified this effect and it may be that at high P/L ratios the effects of alloy addition are of less significance. This would obviously be advantageous in producing an aesthetic restoration. It is also important to bear in mind that in order to be suitable for universal use, this material must be comparable with
amalgam in terms of clinical performance. Therefore the selected comparator was a
disperse phase alloy, in order to compare the performance of the metal reinforced GIC
with a clinically successful restorative amalgam.

The disperse phase amalgam used as the comparator does not have the composition of a
conventional amalgam. Dispersalloy is a high copper alloy with up to 40% of the tin
replaced by copper in the form of micron size silver-copper, eutectic spheres (72% silver
& 28% copper) (Dentsply Ltd, UK). Tin is the major cause of corrosion in all alloys.
Dispersalloy amalgam was selected because of its performance and successful use over
long periods of time. A number of clinical studies have demonstrated the superiority, in
terms of low creep and less corrosion, of a disperse phase alloy over conventional

5.2 Pilot studies:

5.2.1 Compressive strength:

The test regime for compressive strength selected was that proposed in ISO 9917
"Water-based Dental Cement". Some modifications to the procedure were however
investigated in the pilot studies.

The initial study monitored the effect of reducing the conditioning period of the
specimens within the mould at 37°C from 60 min to 10 min. The light cured resin (Hi-
glaze) was applied to the surfaces of the specimens to match the clinical recommendation.

The materials have a relatively long setting time and 10 minutes is close to the earliest
time at which finishing these restorations is appropriate. The result showed that the mean
strength of 6 specimens was lower than that for similar reinforced glass ionomer
materials tested by other workers (Williams & Billington 1991 & Williams et al 1992a),
and the scatter of results showed a variation of 14% which is high in comparison with other workers’ results. The results suggested that 10 minutes is too short a conditioning time to permit this material to achieve a consistent acceptable strength.

It implied that the rate of cement maturation may vary and that the ten minutes conditioning is in fact intruding on the primary setting reaction. Therefore the test method was modified.

The technique was changed in the second study by increasing the conditioning period of the specimens within the mould to 60 min. This evaluated the effect of extension of the conditioning period on the material maturation and was similar to the method prescribed by ISO 9917. The number of specimens were increased to 8 specimens to minimise the influence of outliers on the results. The results of this study showed no change in strength and a wide scatter of results was still noted with a variation of 29.3%. This was due to the substantially lower strength values obtained for two of the specimens. However, no obvious flaws were noted in these two weak specimens prior to loading, and no voids or irregularities were seen in the fragment after loading. It was considered that the wide scatter in the results and the low strength of two specimens were likely to be a result of irregularity on the end surfaces of the specimens.

In the third pilot study the method adopted was as described in ISO 9917 (1991) except that the ends of the specimens were ground flat before removal from the mould. No glaze was applied on the surfaces. The specimen numbers were increased to 15 again to minimise the effect of one or two potential outliers. The result of the study showed substantial improvement in strength and the scatter of results was substantially lower than in the previous studies.

A further preliminary investigation was then set-up to evaluate the effect of application
of resin to the exposed end of the specimen after surfacing the ends of the specimens and conditioning for 60 min. The results showed a reduction in strength. It was apparent that the resin coating adversely affected the results. This may be due to the resin being more elastic than the cement cylinder or uneven surface due to resin coating.

It was decided that the definitive specimens for compressive strength testing would be tested as described by ISO 9917 (1991) apart from the fact that no light cured resin was applied to the specimen ends after conditioning. The problem of moisture sensitivity of the GICs was addressed by placing the specimens in the thermostatically controlled oven at 37°C for 60 min in the split mould. This time period has been considered to be sufficient for the initial setting of these cements to be completed and early maturation achieved (Williams & Billington 1989).

The low mean values of strength noted in some of the pilot studies were due in part to the high coefficient of variation of the results. This may be due to the variation in the specimens construction techniques and also failure to ensure the flatness of specimen ends.

5.2.2 Bi-axial strength (shell test):

A further range of studies attempted to examine the most consistent method of construction of bi-axial flexural strength specimens. The initial study was carried out by packing the material into a split ring mould which was placed between two glass slides. No pressure was used initially, and this resulted in specimens of variable thickness, greater than that of the split mould used. Smooth specimen surfaces were however achieved. Initially the light cured resin (Hi-Glaze) was applied and the specimens were
conditioned for 60 min. The mean flexural strength value obtained was lower when compared to that of other workers (Pearson & Atkinson 1991 & Williams et al 1992a), and there was again a wide scatter of results.

The technique was modified in the second study by using metal plates instead of the glass on both sides of the mould. The mould assembly was clamped to produce better material consolidation. The specimens were conditioned for 10 min and then a protective resin layer was applied to limit the possibility of moisture contamination. This is relatively close to the conditions which occur in the mouth. The results of this study showed that the thickness of the specimens was more consistent but the results showed even more scattering. The mean strength value obtained was lower than that in the initial study. This reduction in measured strength was possibly due to the change in conditioning period or to the variable surface created as a result of the application of resin to the specimen.

In the third study the method was as in the second study except that the conditioning period was extended to 60 min. This was comparable to the conditioning regime used in the compressive strength study. The results showed a higher strength than previous studies with a reduced scatter in results.

Pilot study 4 was the same as the third study except that no surface protection was applied. This study assumed that an hour conditioning period would be sufficient for the material's initial maturation. It also brought the test into line with the compressive strength test. The results showed higher strength than pilot study 3 and a reduced level of scatter.

Pilot study 5 was carried out to investigate the influence of maturation time by storing the specimens for seven days prior to test. The results of this pilot study showed no statistically significance different from the previous strength observed for pilot study 4.
and there was little scatter of results. This indicated that the initial cement maturation appears to have been completed by one hour.

These preliminary tests showed that specimen construction and conditioning played a significant part in the strength values obtained for both compressive and flexural strengths. It was apparent that the method of storage could significantly affect the results. Similarly the surfacing of the end of the specimens to ensure that both ends of the cylinder were parallel appeared to be of critical importance in optimising test results in compressive strength tests. It is apparent also from these results that the cement under test was susceptible to change in environment up to one hour. This is almost certainly related to the rate of set of the material. This was probably as a result of the glass formulation and the acid treatment during the manufacturing process. It does however mean that in clinical use the cements may have a potential weakness in the first hour after placement.

5.3 Methods:

5.3.1 Compressive strength:

The compressive strength specimens constructed were 6 mm high and 4 mm in diameter. This is the smallest specimen where representative values of compressive strength could be obtained. It is usually considered that the cylinder should have a length approximately twice the diameter of the cylinder (Craig 1989). If the specimen is too short, the force distributions become more complicated as a result of overlapping force vectors in the length of the cylinder. There is difficulty in the interpretation of the compression test results because both the mode and plane of failure are variable. Failure
can occur either by plastic yielding core failure (secondary shear forces) or by axial splitting (secondary tensile forces). The mode of failure depends on a number of factors such as size and geometry of the specimens, nature of the material under test and rate of loading. However, the effect of increasing the specimen sizes upon compressive strength of GIC has been investigated and no significant differences were found in the values obtained for the different size specimens (Iguodala-Cole et al 1991) with 6 × 4 mm being the smallest specimens used. It is also beneficial if a specimen can be made from one capsule of material. This can just be achieved with a 6 × 4 mm specimen and thus avoids the need for incremental mixing or double mixing which is a potential source of inter specimen variation.

The measurement of compressive strength appears to produce results which are dependent upon the method of specimen preparation, operator and laboratory variability (McCabe et al 1990). They concluded that compressive strength is not a suitable test for inclusion in a standard, in view of the substantial variability in the results obtained, and that an alternative means of evaluation should be sought. However, compressive strength has some importance in the process of mastication. The use of this test in the present study can be supported as only one operator was involved and therefore as a comparative study the results would be valid. Considerable care was taken to ensure that the variability was minimised.

5.3.2 Uni-axial flexural strength (Four point):

Uni-axial flexural strength evaluation requires a specimen only 2 mm wide and 2 mm high which closely represents the dimensions of a restoration. It may also be
argued that flexural strength more closely represents the type of forces generated in the mouth when the occlusal surface of a restoration is under compression and the base of the restoration is under tension.

Four point loading, as shown in Fig 2, was used with the specimen supported by rollers set 20 mm apart in the lower span. The load was applied to the specimen area between the two rollers in the upper span. The cross head speed was 0.5 mm/min. This speed was selected as a compromise to reduce the influence of slow cracks propagation as well as to avoid a too high fracture rate.

The difference between the three and four point bending tests is the loading pattern which is at the mid point of the specimen using only one central roller in the three point, but using an area between the two upper span rollers in four point test.

The four point application to an area ensures that the forces are well distributed and minimising the risk of specimen distortion or failure along a fault line.

The loading mode at all four contact points is compression whilst the area between two lower span rollers is put under tension.

5.3.3 Bi-axial flexural strength (Shell test):

The shell testing was used as an alternative method of establishing the flexural strength of materials under test. It has previously been used to evaluate dental ceramics which are brittle materials (Sced et al 1977 & Piddock et al 1987 & Ban and Anusavice 1990), whilst Williams et al (1992a) used it to test GICs. Conventional GICs like other dental cements are considered to be brittle materials, and therefore this test method would theoretically be appropriate. There is some variation in opinion as to its suitability for
resin based material, which can show plastic deformation prior to fracture. The current GIC shows little if any plastic deformation during the test regime which confirms that the GIC behaves as a brittle material and this test is appropriate for use.

The advantages of the bi-axial test method are:-

1) It ensures that any edge discrepancies on the specimen do not affect the test results, and allows small specimens to be tested since the annular ring on which the disc rests defines the periphery of the test area.

2) No surfacing of the specimen is required, provided that the specimen is flat and in contact with the annular ring evenly at all points. This can be verified by visual assessment prior to loading and also assessing the number of fragments produced.

3) The maximum tensile stresses occur within the central loading area.

4) The specimen size compares well with that of clinical restorations.

The bi-axial test (shell) is considered to be a more reproducible method of testing flexural strength than either three or four point beam tests. It is because the uni-axial specimen preparation is difficult and any marginal irregularities or defects within the bulk of the specimen, will influence the results and may also be difficult to detect visually (Ban & Anusavice 1990). Ban and Anusavice (1990) also concluded in their study that the bi-axial test is simple to perform and provides a better simulation of the clinical relevant specimen size than that used for the other strength tests (compression test), by making the disc’s thickness similar to that of a small restoration, such as in the class I occlusal restoration. This was confirmed later by Williams et al (1992a).

The use of a split ring when fabricating specimens permitted air and excess material to
escape as the metal plate was lowered on to the specimen. This feature ensured a relatively uniform thickness.

The tracings made by the chart recorder of load against time were used to confirm that the specimens were behaving in a brittle manner (important if shell test theory was to be applied). This was represented by a tracing which rose steeply and then dropped immediately when fracture occurred. The chart tracing was also used to measure the elastic modulus of the materials.

5.3.4 Bonding strength to enamel and dentine:

It was decided that human rather than bovine enamel and dentine would be used to carry out the test, bovine dentine while providing a larger surface area for test, is anatomically not the same as human dentine. There were sufficient human teeth available to carry out the test both on enamel and dentine successfully. Two hundred freshly extracted, non-carious, human molar teeth were initially immersed in 10% formol saline for 10 minutes. This was to kill any pathogens and ensure that there was no risk of cross infection from the teeth during the experiment. While the formol saline will fix organic tissues, it is unlikely to affect the dentine within the crown of the tooth during the 10 min exposure time and any changes which did occur would be likely to be minimal. A more critical factor is the time of test after extraction. This was kept to a minimum. The primary concern relates to degradation of the collagen which form a substantial part of the dentine. Since it has been proposed that adhesion by GIC to dentine is both to hydroxy-apatite and to collagen, then the time after extraction and the pretreatment is important. Changes to the collagen in particular may be significant.
The teeth prepared for dentine bonding were sectioned horizontally at the maximum bulbosity of the crown in order to remove the occlusal portion of the clinical crown and to expose dentinal tubules of a range of size and with different orientations. It was also to ensure that large dentine surface area was exposed and sufficient bulk of dentine was available for adhesion testing.

The exposed dentine surfaces were abraded using silicone carbide paper and water to remove the smear layer, but enamel surfaces were not abraded to maintain the enamel composition and to avoid any possibility of exposing the dentine surface.

Both enamel and dentine surfaces were treated with a solution of 25% polyacrylic acid. It was recommended to partially remove the smear layer and enhance the cements bond to tooth structure (Barakat & Powers 1986, Hinoura et al 1986 & Mount 1994).

One capsule was used for each specimen to reduce any consequences of variation in mixing of the material.

The brass cylinders used in the study were mechanically prepared and care was taken to ensure that their edges were regular and uniform. The cylinder of 3.20 mm (inside diameter) 4 mm height was used in this study, because specimen cylinders less than 3 mm in height may develop stress concentrations within the bulk of the material and away from the interface (Van Noort et al 1989). The mixed cement was condensed into a brass cylinder held against an area of tooth surface. The cylinder was left in place after packing to avoid any stress being created on the specimen and also because it was difficult to remove after the cement had set. The cement outside the confines of the brass cylinder at cement/tooth interface was removed using a dental probe. This was because excess cement beyond the bonding surface area would affect the results.
The most common tests for evaluating the bond strength to enamel and dentine are tensile and shear tests. The direct uniaxial tensile test requires a universal joint system to align the specimen and substrate so that the forces act at right angles away from the specimen surface. However, it is because of stress concentrations, peel and shear forces may occur during the direct uniaxial tensile test leading to false results, the shear test was utilized for the current study. A further problem which has been reported previously with GIC is the cohesive failure of the specimen rather than failure of the adhesion bond. Care was taken to avoid peeling of the bond and to ensure that the bond fails at the plane surface determined by the test, and not in the mode created by characteristics of the materials. Care was also taken to apply the load parallel to the substrate and as close as possible to the bond interface. It is because interfacial stress distribution during shear testing is particularly sensitive to load application. As the distance between the point of load application and the bonded surfaces is increased, non uniform interfacial tensile and compressive stresses developed at the edges of the bond interface. The failure may, therefore, be initiated on the material surface and not at the bond interface.

5.3.5 Erosion resistance:

A series of publications by Kuhn et al (1982, 1983a & 1983b) showed clearly that the dissolution rate of a cement in a small volume of liquid (10-500) is controlled by the volume of liquid and its frequency of change. The process is believed to reach some sort of equilibrium with respect to time, particularly when small volumes of liquid are used. As the solution in which the specimen is immersed becomes increasingly saturated with extracted ions, so the rate of release of ions from the cement slows. The classical test is
also inadequate for cements where the liquid is one of the constituent parts (water and resin based cements). Evaporation of the component can lead to false results. Such conditions cannot compare with the oral environment where a cement is irrigated by saliva which is constantly being removed. Under these conditions the concentration of ions is not allowed to increase.

The jet-test system used a volume of liquid, usually around 6 litres, made up from deionised distilled water and lactic acid. The volume of liquid used is much greater than static tests previously used and this ensures that the ions released into the solution are substantially below that which would lead to slowing of the release and may be considered to be an infinite sink. The solution was made to the required concentration and stored in a polyethylene container for 24 hours prior to use. The solutions were then circulated through the apparatus by pump. Six specimens were arranged in small holders and the liquid emerged from number of small jets impinged on the specimen surfaces, one specimen below each jet. This impinging test also permitted a mechanically disruptive element to combine with the chemical erosion occurring during the test procedure. At the same time turbulent flow is not required otherwise the surface of the cement will be unevenly disturbed.

The test was run for 24 hours and calculations are based on the assumption that the erosion rate of the cement is the same each hour. The time period selected was long enough to ensure that sufficient material was eroded to achieve quantitative measurements. Care was taken to ensure that turbulent flow due to excessive erosion did not occur. This was confirmed by the loss of material across the diameter of each
specimen which was relatively similar. A 24 hours test period was also used by Setchell et al. (1985) who pointed out that cements based on polyacrylic acid erode less than materials based on polymaleic acid. However, erosion rate for other luting and acid base cements are usually substantially faster.

The jet-test temperature used in this study was 37°C. Williams et al. (1994) evaluated some factors influencing the lactic acid jet test and found that the test temperature was much easier to control and also concluded that the 37°C temperature more clinically relevant.

It had previously been found that weighing rather than volumetric dispensing gave more reproducible results (Kuhn et al. 1984). It has been also suggested that erosion should be measured by weight loss which is easier to perform and takes an average measurement rather than relying upon a depth measurement on an unevenly eroded surface (Williams et al. 1994). However, weight loss measurements of different material with different density may lead to differences in results which may prove misleading.

5.3.6 Microleakage:

The many different techniques used in the assessment of microleakage in dental restorations had been reviewed (Shortall 1982, Taylor & Lynch 1992 and Alani & Toh 1997). There is no single satisfactory test that will predict the intraoral microleakage behaviour of materials. Long term monitoring of microleakage can be achieved by using radioactive isotopes usually of calcium chloride (Going 1972 & Alani 1990). However, this is destructive to tooth structure and lacks clinical relevance (Powis et al. 1988). The method is also objective, complex, sensitive and costly (Alani 1990). However, it does
permit detection of minute amounts of leakage.

Microleakage assessment using the dye penetration method was chosen for this study because it provides a simple, relatively cheap, qualitative method of evaluating the potential for leakage around GIC at both the cervical and occlusal margins.

**Cavity preparations design:**

The cavity design included the production of an isthmus width of 1/3 the intercuspal distance and a proximal box width of 1/3 the total faciolingual dimensions interproximally. Mondelli et al (1980) showed a preparation of these proportions compromised the strength of the remaining tooth structure. This design was therefore chosen in anticipation that cuspal flexural would occur during load cycling. It was also chosen to be the worse case scenario used by a practitioner. The preparation was designed to have a depth of approximately 1 mm from the dentine-enamel junction into dentine, the proximal box extended approximately 1 mm below the cementum-enamel junction and the gingivo-axial width of the proximal box was approximately 1 mm from the gingival tooth surface. These dimensions provide adequate material thickness to ensure sufficient bulk of material for strength.

The enamel cavosurface margins were not bevelled as Kempler et al (1976) had demonstrated that butt joints in enamel margins create better sealing in comparison with bevelled cavosurface margins. Certainly as GIC are brittle material, their use in thin section is undesirable. Jacobsen (1984) stated that bevelling of the cavosurface margins transversely exposes the enamel prisms prior to etching creating a deeper etching for improved bond strength with greater interdigitation of the resin into enamel. It also exposes more surface area of enamel for acid etching. This of course is not as significant
with GIC. However, for bonding, the larger the surface area available the better. Boyde (1985) has shown that in the occlusal surface of class I and II preparations, the prisms are exposed transversely with a butt joint.

The disadvantage of the restoration of bevelled cavosurface margins is that it creates a thin layer of material prone to wear and fracture under loads. This was considered to be important with glass ionomer material, because it is considered to be a brittle material. The butt joint cavosurface margin produces a bulk of restorative material at the margins which is more resistant to loads.

**Mechanical load cycling:**

The forces and movements found during mastication are highly complex and difficult if not impossible to reproduce in the laboratory. The peak load used in this study was based on the work done by Fields et al (1986) on *in vivo* occlusal forces. The magnitude of the forces generated during mastication was found to be between 9 to 180 N, as utilized in a number of studies (Anderson 1956, Anderson & Picton 1958, Helkimo & Ingerwall 1978, DeBoever 1978 & Gibbs 1981). The peak load chosen for the current experiment was at the upper end of the range of forces quoted.

The Instron Universal Load Testing Machine used in this study could only produce limited cyclical axial loading. It has a number of limitations which include:-

- It is different from the oral environment where the movement of mastication takes place in a three dimensional plane.

- The overrun on the stepping motor driving the cross head leads to small variation in the load applied, despite a closed loop circuit correcting the overrun at the end of each cycle.

The faster the cross head speed the greater is the overrun.
The cross head speed, in this study, was 25 mm/min. A reduction in the displacement would reduce the overrun.

Load cycling as described by Morin et al (1984) using the servohydraulic testing machine (S.H.T.M.) could have provided a more accurate simulation of intraoral conditions. It is possibly with machine compensation for any relaxation within the tooth structure or its mounting. The advantage of the hydraulic testing machine include also the ability to provide a constant and rapid cyclic load.

As an alternative the buccal and lingual cusps of the teeth were loaded to closely simulate the triangular movement occurred in the mouth during mastication.

Ideally the specimens should be loaded within a few minutes of placement to simulate clinical conditions. This phase of the experiment tested the materials under optimum conditions, the loading being delayed for one hour until the material had been conditioned and matured. This ensured that the bond strength of the GICs to enamel and dentine had been optimised. This was in part due to the time required to set-up the test in the cycling machine and also because the test time was matched to the time used for conditioning in the load testing phase of the study.

**Dye penetration:**

A 0.1\% solution of neutral red dye was used to provide a contrasting colour in the areas where microleakage occurred. Soderholm (1991) suggested that a chemical equal in size to the molecular size of bacterial toxin should be used. Although the dye molecules are considerably smaller than bacteria, they can demonstrate marginal
percolation and leakage which would allow passage of acid and other toxic products of bacterial plaque along the tooth/restorative interface. The dye can therefore demonstrate the potential for undesirable effects of marginal breakdown, secondary caries, tooth sensitivity and pulpal irritation (Phillips 1965). Neutral red dye was selected as its penetrating colour was easier to see against the silver colour of the Hi-Dense when compared with methylene blue.

5.3.7 Storage media:

The storage medium (deionised distilled water) was used for storing all the dispersalloy, EXPT and the Hi-dense glass ionomer materials specimens. The storage medium (artificial saliva) was used at the early stage of this study for storing the Hi-Dense specimens for the mechanical properties tests. Three litres of artificial saliva were prepared, one litre at a time as described previously (Tanni & Zucchi 1967). The pH of the artificial saliva was reduced to 6.7 at the beginning of the experiments by bubbling CO$_2$ through the solution. However, after one week or more it was noted that the pH of the solution was becoming more alkaline. Since it was difficult to control the pH of the artificial saliva at 6.7, the variation in the experimental condition may be expected to be high. It was then decided that all the test specimens will be stored in deionised distilled water alone.

Further the level of alkalinity was such that the saliva substitution had a pH which was outside the range normally found in the mouth. Changes in pH which showed increased acidity would have been acceptable as this would simulate to some extent the variation which occur in the oral cavity.
5.4 Results:

Non-parametric statistical analysis of the results was performed (Mann-Whitney test) because of the relatively small sample sizes. Also the median values obtained in many groups were substantially different to the mean calculated for the same group. This situation suggests the existence of outliers and the Mann-Whitney analysis considering ranking not magnitude of results ensured that the outliers do not skew the analysis.

Mechanical properties:

The mechanical properties of the cements as evaluated were very variable. The results showed that the strengths of all the materials including the disperse phase amalgam (the clinical comparator) increased with time. This is not unexpected as all the materials including the amalgam mature. The amalgam strength was slightly greater than expected. The maturation of GICs is variable, initially GIC has a relatively low strength. Three different patterns of strength increase have been proposed:-

- an immediate increase in strength within the first 24 hours continuing to rise and then stopping at a certain point (Crisp et al 1976a).

- an increase in strength which continues for several months before being maintained (Williams & Billington 1989 & 1991 and Pearson & Atkinson 1991).

These two patterns were attributed to the continuous formation of polysalt complexes during an ongoing acid-base reaction (Crisp & Wilson 1974) or to the slow rate of cement maturation (Crisp et al 1976a, Simmons 1983 & Williams et al 1992a).

- an immediate increase in strength over the first 24 hours which is then maintained before degradation (Smith 1968 & Nicholson & Abiden 1997). This is possibly attributed to the hydrolytic instability (Crisp et al 1976 & Smith et al 1988). Nicholson and Abiden
(1997) demonstrated that it appeared to be attributed to the strengthening of the silica gel due to a high cross-link density which makes the material more brittle and subject to failure rather than elastically deform.

5.4.1 Compressive strengths:

The disperse phase amalgam showed significantly higher early compressive strength within the first 24 hours than the two glass ionomer materials used in this study. The amalgam maintained its strength at six months. There were no significant differences between Hi-Dense and EXPT at all test periods apart from one month, where EXPT was stronger. The rate of maturation of the metal reinforced material was slower than the EXPT, the peak value for Hi-Dense being achieved at three months whereas the EXPT was stronger after one month. Both materials maintained their strength thereafter. These contradicted the results of Coury et al (1986) and Beyls et al (1991). They have showed that the addition of such relatively low metal content (20% alloy) improved the compressive strength of glass ionomers to a certain extent. These results suggest that the use of high powder/liquid ratios for cements can enhance mechanical properties adequately without addition.

There appeared to be no decline in compressive strength for either material with respect to time. This is in keeping with the results of Williams & Billington (1991) who found that over their investigation period of 24 hours to 4 months, glass ionomer restorative materials based on polyacrylic acid maintained or even showed a slight increase in compressive strength, only those based on copolymers of acrylic acid initially increased in strength, but thereafter showed deterioration.

The compressive strength obtained for the EXPT material which has a very high powder
liquid ratio in comparison with most currently available commercial material was of interest. The value was rather higher than expected. It would be of interest to carry out similar long term tests on more recent material such as Ketac Molar where the P/L ratio has been raised. On the evidence of the compressive strength test alone the enhanced P/L ratio of the EXPT material has nullified the effect of alloy addition.

5.4.2 Uni-axial flexural strength (four point):

The disperse phase amalgam showed high early flexural strength which was maintained at six months. It is well known that the early strength of conventional GICs is low compared to that of other restorative materials (Crisp et al 1976a, Simmons 1983 & Williams et al 1992a). This may be attributed to the slow rate of cement maturation. The Hi-Dense and EXPT were of similar flexural strength during the initial stage of the experiment. This was not surprising as the glass for both materials was derived from the same source, with a similar particle size distribution. The peak value of strength for Hi-Dense being achieved at three months whereas the EXPT showed a slight increase in strength over the first six months but there was no significant difference between the various test interval values. Hi-Dense was significantly stronger than EXPT at later time periods. Both materials exhibited early strengths which were higher than those of many other conventional GICs or cermets (Williams et al 1992a). The pattern of results differs from the results in compression where no significant differences were observed between the materials. Glass ionomers based on polyacrylic acid were also found to have a higher flexural strength than those based on a (maleic/acrylic) copolymer (Pearson & Atkinson 1991). Multiple comparisons of strength at different time periods indicated that the initial four
point bending flexural strengths of the EXPT was maintained whereas the Hi-Dense increased and then maintained over three months. One of the concerns about alloy addition is that the alloy may not take part in the reaction remaining as an inert filler. This would then lead to a potential for fracture to occur at the interface between cement and alloy particles potentially weakening the cement. However, the mechanism by which the alloy powder reinforces the cement is not clear. It has been suggested that the metal particles participate in the setting reaction, the polysalt matrix is chemically bond to the superficial oxide layer on the alloy particles (Hotz et al 1977 & Kerby & Bleiholder 1991).

Both cements had a high powder/liquid ratio which confirm the suggestion that higher strength is achievable when a high powder/liquid ratio is used. A stronger polysalt matrix may also be the result of a high molecular weight polymeric acid in the liquid formulation. The slightly lower P/L ratio used in the EXPT material may will explain the slightly lower mechanical properties here.

5.4.3 Bi-axial flexural strength:

Hi-Dense cement was generally stronger than its counterpart at later time periods. The strength was maintained for Hi-Dense where as EXPT showed a small but significant decline at three months. The peak value for Hi-Dense was achieved at three months whereas the EXPT was after one month.

It has been reported that the conventional GICs experience a steady build-up in strength which can continue for several months, using two different test, uni-axial test used by Pearson & Atkinson (1991) and compressive strength test used by Williams and Billington (1991). This has been attributed to the continuous formation of polysalt complexes during
an ongoing acid-base reaction (Crisp & Wilson 1974).

It is however, interesting to note that the bi-axial strength of the EXPT shows a significant decline after three months. This is possibly attributable to hydrolytic instability as reported by Crisp et al (1976) and Smith et al (1988). A similar decline was also observed by Williams & Billington (1989 & 1991) and Pearson & Atkinson (1991) and they thought this may have been due to hydrolysis of the matrix. Recently however, Nicholson and Abiden (1997) demonstrated that a similar decline occurs when specimens were stored in dry air or vegetable oil. The decline in this work is unlikely to be due to hydrolysis. Rather they suggested that it appeared to be a consequence of the higher cross-link density that can develop in these cements. A higher cross-link density would make the material brittle and hence more sensitive to faults and flaws. This explanation would also apply to the acrylic/maleic acid copolymer cements.

The variation between EXPT and Hi-Dense may be in part due to the alloy addition which has been previously thought to improve the mechanical properties (Williams et al 1992a).

The result of this study showed that the strength of Hi-Dense was either maintained or increased over six months, while EXPT showed strength improvement during the earlier phase of the experiment but subsequently a statistically significant decline in flexural strength was observed at three months.

The results showed that there were no significant differences between uni-axial and bi-axial tests except at the one day period for Hi-Dense, and at one day, one week and one month for EXPT where bi-axial strength was significantly higher than uni-axial
strength. There appears to be some benefit in using the bi-axial (shell) test for testing flexural strength rather than uni-axial (four point) because it is a simple test to carry out. It is economical and the specimens are easy to construct. The results are generally higher and it could be argued that these show the materials in a better strength. However, the reduction in the scatter of the results may have contributed to this. Further, the reduction in the risk of specimen defects, more common occurring in the uni-axial tests, means that the material is under test rather than operator techniques.

5.4.4 Bonding strength to enamel and dentine:

GICs have been shown to bond to tooth structure containing a high hydroxyapatite content (inorganic) (Hotz et al 1977, Coury et al 1982 & Wilson et al 1983). In this study bond strengths to enamel were substantially greater than those to dentine which is in accord with other workers (Hotz et al 1977 & Wilson et al 1983). This suggested that the mode of bonding is principally to the inorganic phase (hydroxyapatite) (McLean 1980), although in vitro studies have shown that GICs do adhere to collagen (Beech 1973).

The bond strengths to enamel and dentine of Hi-Dense and the EXPT tended to be substantially higher when compared to those reported in the dental literature for conventional GICs 2.5 to 5.3 MPa to dentine (Hotz et al 1977 & Kerby 1992) and 3.8 to 9.3 MPa to enamel (Hotz et al 1977, Coury et al 1982 & Kerby 1992).

The bond strengths appeared also to be relatively similar to the alloy reinforced GIC (Miracle Mix) and higher than cermet which have been tested by Kerby (1992), who related the higher bond strength to dentine to the location of the site for adhesion testing on dentine, which was a substantial distance from the pulp.
The long term bond strengths of Hi-Dense and the EXPT appeared to be higher than the early bond strength observed. This may be due to ongoing maturation of the cements which has an enhancing effect on the ionic bond at the bond interface. It may also be that the ion exchange which is known to continue may indirectly lead to further interaction between tooth and GIC matrix.

The difference in the test methods, the materials used, the materials molecular weight and the powder/liquid ratio may contribute to certain extent in the results.

The mode of bonding failure was predominantly adhesive for dentine and cohesive at the bond interface for enamel. This may indicate that moisture incorporation during the early stage of setting of the cement is associated with bonding to dentine, producing a weaker material at the interface. The higher organic content (collagen) of the dentine may also adversely affect the bond strength (Aboush & Jenkins 1984). It seems probable that the rate of build-up of strength of the bond would be slower to collagen. It may also be that the high P/L ratio may lead to a material which does not wet the surface of the dentine as well.

When cohesive failure at the bond interface occurred with the enamel at later periods, higher loads were involved which may explained that the glass ionomer materials maturation continue for long period of time before reach the full strength.

When cohesive failure occur the absolute bond strength values have not been measured. Again the influence of maturation rate may be of significance. Cements which have a rapid set may well not show this progressive increase in bond strength as the maturation is faster. Again the influence of the glass and the pretreatment may play part in this.
The metal reinforced GIC and EXPT had significantly higher bond strengths to enamel which increase with time and bond strengths to dentine which were maintained over the extended time periods.

5.4.5 Erosion resistance:

The results generally showed that measuring weight loss produces a ranking order for values which was similar to the results from measuring the average erosion depth for both EXPT and Hi-Dense materials. This is in accord with the work done by Wilson et al (1986) and Williams et al (1994). However, after six months storage the rate of erosion of the two materials was not substantially different. This is particularly the case with the EXPT material when measuring the erosion depth. This highlights the problem of comparing materials with different densities. Under these circumstances the weight loss data gives a rather misleading impression of the relative loss of each material.

The weight loss of EXPT was highly significant greater than Hi-Dense at all testing periods. The erosion rate of Hi-Dense at 24 hours was 0.08 mg/h which is less than any conventional or reinforced glass ionomer cements previously tested (Billington et al 1992). Furthermore, Hi-Dense has a lower erosion rate than glass ionomer cements containing radiopacifying elements which have been tested by Williams et al (1993). The erosion rate of both materials decreased when stored in deionised distilled water for long periods of time. This demonstrated that both glass ionomer materials tested show a significant improvement in erosion resistance with time. This has not been observed for other dental cements. This is in accord with the work done by Williams et al (1992b). They tested the effect of maturation on the erosion of glass ionomer and other dental
cements and found that cements using polymeric acids had erosion rates that reduced with time. The marked improvement in erosion resistance after six months storage particularly of the experimental material again tend to imply that the cement maturation is a critical component in the prediction of its longevity.

The metal reinforced GIC (Hi-Dense) has a low initial erosion rate when compared with the experimental material and therefore good erosion resistance compared with other types of water-based dental cement. This could be of importance in evaluating clinical performance.

5.4.6 Microleakage:

Microleakage is of considerable importance in the evaluation of a material for clinical suitability. It potentially demonstrates a line of least resistance for degradation to occur in the longer term. Even though the technique involved a destruction, provided a number of sections are obtained, a view of the degree of leakage can be estimated. The technique itself does not only evaluate the material but to some extent also the clinical condition for use, as the materials are packed in to cavities using normal clinical methods. In order to obtain as much information as possible the teeth are sectioned to produce the largest practical number of surfaces. This in most cases is limited to four cuts bucco-lingually and one through the mesio-lingual aspect. Each surface is scored as the curf of the blade means that facing surfaces are at least 0.28 mm apart after sectioning.
It was decided that the occlusal and cervical margins leakage could not be compared quantitatively. Their scoring was not comparable either, because of the absence of the enamel cervically, or the difference in the specimen numbers between the two margins.

Qualitatively, the results showed that the dye penetration was greater at cervical margins rather than at occlusal margins for both glass ionomer materials. This has previously been observed by Yap et al (1996). The EXPT showed significantly more dye penetration at one day and one month cycling than Hi-Dense at the cervical margin. Hi-Dense showed the highest number of 0 scores (91.88%) at one month cycling compared with EXPT which showed (75%) 0 scores of unloaded specimens at the occlusal margins. The results obtained could be expected to be different if the restorations were themselves loaded rather than the opposing buccal and lingual cusps of the teeth, because restoration fracture would have been possible. None of the specimens fractured despite the relatively high load selected.

Mechanical load cycling had no significant influence on leakage patterns at the cervical margins of either material nor at the occlusal margin of the EXPT material. This is possibly due to the dimensional stability of GIC, and is in agreement with the results obtained by Darbyshire et al (1988), using a servohydraulic testing machine. The dye penetration at the occlusal margins for Hi-Dense at one month cycling was reduced. This may be due to material maturation. However, a number of studies by Jorgensen et al (1976) and Erickson & Jesen (1986) have reported an increased leakage with mechanical load cycling.
The metal reinforced GIC (Hi-Dense) showed less leakage than EXPT at both cervical and occlusal margins.

The bond between Hi-Dense and tooth structure like all glass ionomer cements is ionic in nature (Wilson et al 1983). Enamel has a higher inorganic content when compared to dentine and cementum and this may explain the greater number of 0 scores obtained at the restorative/enamel interface as compared to that at glass ionomer-dentine/cementum interface. Certainly the results of the adhesion study carried out here tend to support the microleakage finding.

The improved results with Hi-Dense may also be related to material handling. The Hi-Dense is condensable and appeared to wet the surface of the tooth quite well. It is not clear whether this applies to the EXPT material and certainly on the evidence of this study, adaptation of the high P/L conventional material is not quite as good.

5.5 Evaluation of laboratory studies:

It is apparent that neither of the glass ionomer cements have the same mechanical properties as the dispersed phase alloy. In particular the compressive strength of the amalgam is substantially higher. However the early compressive strength is relatively similar to composite resins which are used in selected sites in the posterior segments of the mouth. This suggests that these materials may have some use in selected sites.

A comparison of the properties for the two cements shows a number of interesting variations. The mechanical properties of the two cements are not very dissimilar. Over the extended time intervals for which tests were carried out Hi-Dense does appear to perform marginally better. However the differences observed here were substantially less
than those reported by Williams et al (1992a). In that case however the conventional GIC was mixed at a much lower P/L ratio. This implies that if similar P/L ratios are used the addition of alloy does not greatly increase the mechanical properties of the materials. There is however a distinct difference with respect to time in that the EXPT material reached its peak strength in advance of Hi-Dense and there followed a slow decline which was not observed with the metal reinforced counterpart. This does indicate that the alloy may play some part in reinforcing the material and may counter the effects of either the cross linking of the silica matrix suggested by Nicholson and Abiden (1997) or the water degradation to which these materials are prone. The former is probably the most likely but the mechanism is not clear and further work in this area is required.

The erosion resistance of the two cements again favours the alloy-reinforced material but in this case by 6 months there appears to be little difference between the two. It is however important that the early erosion is low for clinical purposes and here the Hi-Dense is substantially better. This implies a more rapid first phase maturation. This may be due to the proposed interaction between the oxide layer on the alloy particles and the acid that would provide an early matrix that is quite strong and would be more resistant to erosion.

The adhesion data tends to favour the EXPT material. This is not surprising in that the alloy will have an affinity for the acid and compete with the calcium. It is also interesting to note that the bond strengths obtained were higher than those reported elsewhere. This may be due to the test method but may also be associated with the use of mechanically stronger cement, which are less likely to fail cohesively. What is clear is that the values for adhesion of GICs do not represent, in the vast majority of cases, the ultimate bond strength of these materials to tooth structure. This does beg the question as to whether
the use of bond strength values is correct in the assessment of adhesive performance. This is an area, which require much further detailed investigation.

Marginal leakage is one parameter which can be used to assess the performance of the material and also has been suggested as a means of assessing the level of adhesion. This is probably not a direct guide to adhesion but more a means of assessing the adaptation of the material to the cavity. The handling properties of the material and also the viscosity of the material frequently influence it. It is interesting to note that the Hi-Dense material is of a putty like consistency and does not appear to wet the surface of the dentine. However, it is condensable and can be plugged into the cavity with conventional amalgam pluggers. The EXPT material while being much stiffer was not quite so easily condensable and this may account for its slightly inferior performance in comparison with the alloy addition material. Both materials did however perform well in this test and even under simulated chewing did not appear to break down.

On the evidence of these tests although the alloy addition material did not show such a substantial increase in mechanical properties as had been expected, its better performance in a number of other tests indicated that in the longer term it might perform better than the high P/L ratio conventional GIC.

Hi-Dense was therefore selected for a preliminary field trial evaluation to assess its performance in a general dental practice. In the light of previous experience with GICs particular attention was paid to the wear.
5.6 Occlusal wear evaluation:

In evaluation of occlusal wear, the Hi-Dense capsules were mixed for 15 seconds in a mechanical mixer (Degussa Dentomat 2) using a capsule extension adaptor to increase the mixing speed. This mixer was oscillating at 2800 cycle per minute which was lower in comparison with Silamat Plus (Vivadent) mixer oscillating at 4500 cycle/min, without the need for capsule extension adaptor. While this provide an improved mixing speed, it has been found that these materials are not so well mixed using an extension arm. However, the Dentomat is the most commonly found mechanical mixer in the UK.

There was a wide range in wear values for the duration of the trial. The range of dispersalloy amalgam wear was from 25-250 \( \mu m \) after 12 months. However, the range of Hi-Dense GIC wear was greater ranging from 50-500 \( \mu m \) after 12 months. The results in this study are not strictly comparable to those of other investigations.
The wide range of values may indicate that factors other than the composition of the material may have contributed to the extent of the wear. Taylor et al (1989) have shown that restorations in first and second molars wore more readily than premolars. Taylor et al (1990) measured the indirect wear of composite using two different calibration standards viz. the Moffa Lugassy Standard and the Leinfelder Standard. They found that after 5 years the mean wear using the Moffa Lugassy standard was 99±44 \( \mu m \) and the Leinfelder value was 191±43 \( \mu m \).
A multi-centre trial measured the direct wear of posterior composite resin (Occlusin), using the Leinfelder Standard showed a slightly different wear values. The mean values for the occlusal wear was 76 \( \mu m \) after three years (Norman & Wilson 1988), and 188 \( \mu m \)
after five years (Wilson & Norman 1991). Another trial has used the same method of measurements have also shown different mean value, 154 μm after five years (Wilson et al 1988).

The current study utilized the Ivoclar Vivadent standard and the mean value of wear of dispersalloy was 27.1 μm for upper arch and 38.2 μm for lower arch after one year. However, the mean value of wear of Hi-Dense was 209 μm for upper arch and 240.8 μm for lower arch after one year. There were no significant differences in occlusal wear between upper and lower teeth.

Both the Ivoclar Vivadent and Lienfelder standard are replicas of selected actual restorations. Moffa Lugassy standards are not tooth shaped but circular cylinders with flat bottomed circular depressions of known depth.

5.6.1 Factors affecting the performance of the Hi-Dense GIC:

There were a number of factors which may have affected the performance of the reinforced glass ionomer cement:-

1) Material variation:

Both dispersalloy amalgam and the Hi-Dense materials are encapsulated and reported to be condensable, but the Hi-Dense material is slightly plastic and requires greater care during packing. It is also unsuitable for carving so that initial contouring must be carried out with a flat plastic instrument. Once the material had hardened, it is required to be trimmed with engine instruments. This presents greater difficulties with contouring and can lead to small marginal discrepancies. It will also create a relatively rough surface in certain areas. The use of a protective glaze would certainly have influenced the initial wear assessment since the glaze will not remain in situ for a period
in excess of one week. Thus, the wear over the first six months will, in part, be due to loss of resin and partly due to wear. The wear between 6-12 months will be entirely due to material loss.

2) Tooth selection:

The variability of tooth position, angulation and rotation would inevitably result in some difference in wear due to variation in occlusal contact and load. Size and shape of cavity is also of importance since there is substantial evidence that cavity width influences wear rate (Wilson et al. 1986).

3) Operator variable:

The placement was carried out by one operator in a busy NHS practice. This is probably the worse case condition for evaluation. However, to be acceptable as a restorative material, any restoration must perform satisfactory. The use of one operator does immediately produce a bias, in that any idiosyncratic placement and manipulation technique can influence the trial outcome. The operator, apart from being briefed as the necessary precautions to be taken, was asked to develop his own technique. A more prescriptive clinical trial is in progress and preliminary results (Pearson 1998) suggest that the material is much more technique sensitive than was previously thought.

4) Assessor variable:

The number of assessors may influence the wear results, specially when two or more clinicians were involved in the models assessment. Two clinicians were involved in the current study and had previously been trained to assess any defects to estimate the
loss of material. The training was carried out by asking each examiner to score the same number of stone replicas independently. The examiners then sat together to compare their scores and any obvious discrepancy were discussed and agreed. They repeated the same method until they were in good agreement before carrying out the actual scoring for the main study. Even with this level of calibration, there were a number of discrepancies which required further reassessment of a number of the trial models. It would seem advisable that training is carried out before each assessment to ensure calibration has been achieved.

The result of this study has shown that the metal reinforced GIC (Hi-Dense) wore at a greater rate than disperse phase amalgam alloy (dispersalloy). No fracture or marginal failure were observed for either group and all restoration were considered to be clinically acceptable. Year data is relatively short but has been suggested as a screening period after which long term performance can be assessed. Using this, the Hi-Dense in its current form has a high rate of wear and would not be acceptable as an amalgam replacement. It has been suggested that the defect in the material at present time is the problem of protection during the period of initial maturation. This may will be a limiting factor and is likely that substantial work is required to overcome this.

Summary:

The clinical performance of this material indicates that it is susceptible to wear. This wear appears to be at its greatest during the first six-month period but does continue in the subsequent six-month test period. It is quite clear that the performance does not match that of the disperse phase alloy. However, it is fair to say that this material was
used in a number of particularly heavy load bearing area and in that respect has performed reasonably. The evidence to date suggests that the handling of the material at placement is critical and that the isolation form moisture contamination as carried out in the conditioning period for the mechanical tests in this study needs to be strictly adhered to. This makes the handling of these materials substantially more critical when used in the posterior sextants of the mouth. Further work is necessary to provide a simple way or protecting the material during its early maturation phase if these materials are to be considered as a substitute for amalgam even in selected sites in the posterior sextants of the mouth.
Chapter (6)

Conclusions
6 Conclusion:

Within the limitations of this study the following conclusions may be drawn:

1) The long term strength of Hi-dense is either maintained or increased over long periods.

2) The addition of the alloy powder causes a significant increase of the long term flexural strength of the GIC.

3) The bi-axial flexural strength measurements appear to be more discriminatory when compared to compressive or four point flexural strength measurements.

4) The alloy reinforced GIC had significantly high bond strengths to enamel and dentine which were either increased or maintained over extended time periods.

5) The alloy reinforced GIC (Hi-Dense) has a low erosion rate and appears to have better erosion resistance when compared with other types of water-based dental cement.

6) Mechanical load cycling had no significant influence on the leakage pattern both occlusally and cervically for the two glass ionomer materials.

7) The Hi-Dense showed less leakage than EXPT at both margins and it may be clinically acceptable.

8) The Hi-dense wore at a greater rate than disperse phase amalgam alloy (dispersalloy).
Chapter (7)

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  in posterior teeth.
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Acknowledgement

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I would like to thank my cousin Dr. S. M. Hussein, colleagues and all friends, who encouraged me no end, and without their ideas and morale support this work would never have become a reality.
Publications and Presentations

  Evaluation of the long term strength of an alloy-reinforced glass ionomer cement.

  Influence of alloy powder addition on the strength of glass ionomer.

Presentation (Posters):

  Evaluation of the long term strength of an alloy-reinforced GIC.
  British Society for Dental Research, (BSDR), (Abstract No. 378), Bristol (1996).

  Long term in vitro evaluation of the adhesion of an alloy-reinforced GIC.

  Influence of alloy powder addition on the strength of glass ionomer.
  British Society for Dental Research, (BSDR), (Abstract No. 429), Brighton (1997).
Appendices
Appendices:

Appendix 1: Compressive strength of GIC (MPa) (Artificial saliva storage).

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Appendix 2: Uni-axial flexural strength of GIC (MPa) (Artificial saliva storage).

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Appendix 4: Statistical analysis of data.

The all cases test was carried out using non-parametric Mann-Whitney test.

NS = not significance. NS ≥ 0.051
S = Significance. S ≤ 0.05 ≥ 0.011
HS = Highly significance. HS ≤ 0.01
## Statistical analysis of Compressive Strength of Hi-Dense.

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Statistical analysis of Shell Strength of Hi-dense.
**Statistical analysis of shear bond Strength of Hi-Dense.**

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Statistical analysis of Compressive Strength of glass-ionomer cements.

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<td>S (0.021) *</td>
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| 6Months HD | * | * | * | * | H.S (0.0002) * | *
| Year HD  | * | * | * | * | * | H.S (0.0028) |
## Statistical analysis of shear bond strength of glass-ionomer cements.

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### Statistical analysis of erosion resistance of glass-ionomer cement.

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Evaluation of long term strength of an alloy reinforced glass-ionomer cement.
M. A. B. ABOUR*, R. LABELLA, G. J. PEARSON. (Biomaterials Department, Eastman Dental Institute, London).

This *in vitro* study compares the long term strength of an alloy-reinforced (Hi-Dense) with unreinforced glass-ionomer cement (Hi-Fi) using bi-axial and four point bending flexural strength tests, and compressive strength test. Five groups of ten specimens were constructed for each test for each material and allowed to set in a thermostatically controlled oven at 37°C for 60 minutes. The tests were carried out after storage in distilled water for one day, one week, one month, three months and six months. The specimens were loaded to failure in a universal load testing machine and the strength of each specimen was calculated. The results showed that the bi-axial flexural strength test was not significantly different between materials at one day and one month storage time. The Hi-Dense strengths were significantly higher than Hi-Fi strengths at one week, three months and six months storage time. The four point test showed no significance difference at one day time. However, the Hi-Dense strengths were significantly higher at all other times. The Compressive strength showed no significance difference between both materials at all the time period except at one month where Hi-Fi was significantly higher.

It is concluded that the strength of the alloy reinforced glass-ionomer cement can be maintained over extended time periods but the strength of unreinforced glass ionomer cement degraded after approximately one month.
Long term *in vitro* evaluation of adhesion of an alloy reinforced glass ionomer cement.

M. A. B. ABOUR, R. LABELL and G. J. PEARSON. (Biomaterials Department, Eastman Dental Institute, London).

This study evaluated the shear bond strength of an encapsulated alloy reinforced glass ionomer cement (Hi-Dense) to human enamel and dentine. Eighty freshly extracted, non-carious molar teeth were cleaned with an ultrasonic scaler, immersed in 10% formal saline for 10 minutes, washed and then stored in distilled water at 4°C until use. The teeth were divided into eight groups of ten, four groups were prepared for dentine bonding and the other four for enamel bonding. Teeth prepared for dentine bonding were sectioned horizontally at the maximum bulbosity of the crown with a rotary diamond saw to remove the occlusal portion of the clinical crown. All the roots were also removed by sectioning. The sections were embedded in acrylic resin using cylindrical moulds (11 mm diameter x 15 mm depth), leaving either an enamel or dentine surface exposed. These were polished using 500, 800, and 1000 grit silicone carbide paper and water. All the specimens were kept in distilled water at 37°C until the bonding of the cement was carried out. Each tooth surface was conditioned for thirty seconds using 25% polyacrylic acid. The alloy reinforced cement was then mixed in a mechanical vibrator for 15 seconds. The mixed cement was then condensed into a brass cylinder, 3.20 mm (inside diameter) x 4 mm (depth), against an area of tooth surface. The material was allowed to set at 37°C for one hour then stored in distilled water for one day, one month, three months and six months, (ten specimens each), until testing. The specimens were inserted into a custom made stainless steel jig and tested at the loading rate of 0.5 mm/min in universal load testing machine [Hounsfield H25K, Croydon UK]. The load required to induce bond failure was recorded. The shear forces were calculated using the formula:-

\[
S = \frac{4F}{\pi d^2}
\]

Where \(S\) is the bond strength in MPa. \(F\)=Displacement load, \(d\)= diameter of specimen \(^2\). The results are presented in table I. Bond strength to dentine initially was recorded at 4.8 MPa but increased with respect to time and the highest value was recorded at six months (7.6 MPa). Bond strength to enamel was substantially higher (11.7 MPa) initially. Again there was an increase with respect to time to 12.4 MPa. These bond strengths are higher than those noted by other workers who carried out the measurements in tension. The higher bond strength to enamel is in accord with other workers. It is concluded that the alloy reinforced glass-ionomer cement had significant high bond strengths to enamel and dentine which were either increased or maintained over extended time periods.
Influence of alloy powder addition on the strength of a glass-ionomer cement.

This *in vitro* study compares the early and long term strength of a metal admix glass-ionomer cement (Hi-Dense, Shofu) with that of the same material without metal addition (EXP). Both materials are characterised by a high powder/liquid mixing ratio. Bi-axial and 4 point bending flexural strengths and compressive strength were tested. Five groups of ten specimens were constructed for each test for each material and allowed to set in a thermostatically controlled oven at 37°C for 60 minutes. The tests were carried out after storage in distilled water for 1 day, 1 week, 1 month, 3 months and 6 months. Results in table below are mean strengths in MPa (standard deviation).

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<td>51 (5)</td>
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<td>51 (10)</td>
<td>39 (7)</td>
<td>56 (4)</td>
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<td>254 (18)</td>
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<td>42 (3)</td>
<td>50 (6)</td>
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<td>3 months</td>
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<td>47 (8)</td>
<td>55 (6)</td>
<td>48 (4)</td>
<td>283 (23)</td>
<td>288 (18)</td>
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<tr>
<td>6 months</td>
<td>57 (7)</td>
<td>49 (8)</td>
<td>55 (3)</td>
<td>43 (4)</td>
<td>285 (21)</td>
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All results were analysed for statistical significance (p<0.05) using parametric tests. Pairwise comparisons at each time period revealed that Hi-Dense and EXP were equally strong at 1 day. However Hi-dense was generally stronger than EXP at later time periods, except in compression mode when no significant difference was found. Multiple comparisons of strength at different time periods indicated that the initial 4 point bending flexural and compressive strengths of both materials were maintained or increased over 6 months. Bi-axial flexural strength was maintained for Hi-Dense, whereas EXP showed a small but significant decline at 6 months. The addition of the alloy powder causes a significant increase of the “long term” “flexural” strength of the glass-ionomer cement.
### Errata

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Page 160: Table 22: Young's modulus of glass ionomer cements (GPa).

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