



Some Aspects of *In vitro* Testing of Glass Ionomer Cements

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A research report submitted as a partial requirement for the degree of Master of
Dental Surgery in Conservative Dentistry

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September 1997

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Abstract

Glass ionomer cement (GIC) restorative materials have been limited in their clinical use because of their relatively inferior physical strength compared to composite resin (CR) restorative materials. For both CRs and GICs, the International Organization for Standardization (ISO) set standardised tests, but they differ in the standard strength testing for each material. For CRs they specify the flexural test (ISO4049:1988) while for GICs, they require the compression test (ISO 9917:1991). These differences make it difficult to compare CRs and GICs on the same scale. Furthermore, the recent introduction of a resin component into GICs has added more confusion to the assessment of these materials. In addition, the strengths of GICs reported from various physical strength tests may be artificially low due to inadequate handling conditions.

In the first part of this study, the CR and GIC specimens were stored in various conditions and the "shear punch tests" (Roydhouse, 1969, 1970; Smith and Cooper, 1971; Mount *et al.*, 1994 b; Mount *et al.*, 1996) were carried out to compare the strengths of a range of currently available CRs and GICs. A modified technique based on the methods described by Mount *et al.* (1996) was used in this study. Brass washers approximately 18 mm in diameter and 0.8 mm thick were used for the specimen moulds. Each washer had a 6 mm diameter hole into which the materials were placed. A total of 120 (ten specimens each of the material) specimens were made. After storage in water for one week, the specimens were ground and the thickness was recorded before testing. The specimens were then placed onto the Hounsfield Tensometer with a 3.2 mm diameter punch and corresponding die. The punch was advanced through it in a compression cage with a cross-head speed of 2.0 mm per minute. The load at fracture of each specimen was measured. The mean strengths and standard deviations were calculated among the specimens of the material. The shear punch test appeared to be advantageous in allowing the comparison of the physical strength of both CR and GIC restorative materials. This technique-insensitive test can be considered as an alternative for the present standard tests, giving one strength test for assessing a range of restorative materials. Under the experimental conditions considered, GICs showed lower strengths than CRs ($p < 0.05$). The ranking of the strength of the materials were assessed as:

Z-100 > Pertac Hybrid > Silux Plus > Degufill H > Degufill M > HiDense > Fuji IX > Fuji II Cap > Fuji II LC > Vitremer > Fuji II Blue > Miracle Mix

In the second part of this study, the wear resistance and the shear punch strengths were investigated in three GICs (Fuji II Cap, Fuji IX, Fuji II LC; GC Corporation, Tokyo, Japan). These materials were particularly selected to assess the application in the practice of the A.R.T. techniques (Frencken *et al.*, 1994). In this case, GICs are expected to serve as "long-term temporary" restorations, therefore knowledge of these two properties is clinically beneficial. Standardised GIC restorations were placed into cavities prepared in polymer mounting blocks, sealed with mylar strips during initial setting, and stored in distilled water at room temperature for one week. A series of 5 specimens of each material were then rubbed against human molar enamel using a wear testing machine developed at The University of Adelaide. Specimens were subjected to 10,000 cycles of simulated wear under a load of 9.95kg with running water as a lubricant. The wear of each specimen was determined by measuring weight loss. Under the experimental conditions considered, the materials wore at the following rates:

Fuji IX < Fuji II LC < Fuji II Cap ($39 < 43 < 68 \times 10^{-2}$ mg/1000 cycles, respectively)

The shear strength of these three materials was also compared. Fuji IX appeared to have the most appropriate characteristics for the application in the A.R.T. technique.

In summary, this study investigated the physical strengths of some GICs and CRs using the "shear punch test". Also, the strengths and the wear resistance of the three GICs were assessed. From the results, conclusions could be made about the appropriate use of these aesthetic restorative materials.

Declaration

This work contains no material which has been accepted for the award of any other degree or diploma in any university or other tertiary institution and, to the best of my knowledge and belief, contains no material previously published or written by another person, except where due reference has been made in the text.

I give consent to this copy of my thesis, when deposited in the University Library, being available for loan and photocopying.

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September 1997

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Acknowledgement

I am grateful for the opportunity to thank those who have directly and indirectly contributed to my research report as the part of the MDS course at the University of Adelaide in 1994 and 1997. This report could not have been completed without the following people who supported me.

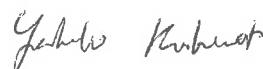
First of all, Drs. Lindsay Richards, Professor Tilly Peters and John Kaidonis as kind, supportive and encouraging supervisors, and Drs. Roger Smales, Owen Makinson, and Graham Mount for their thoughtful advice drawn from their wide experience. Also, many thanks to Michael Willsen who was doing a vacation research program during his fourth year at the Adelaide Dental School with whom I collaborated in the preliminary experiments and whose discussions and suggestions contributed to the wear testing designs and protocols.

Secondly, many thanks to Miss Lyn Waterhouse and Mr. John Terlet for their technical help in using the Scanning Electron Microscope.

Thirdly, heartfelt thanks to Mr. Ray Peterson in the Advisory Center For University Education in the University of Adelaide for his warm, supportive help with English as a second language.

Finally, great thanks to my partner Alister Smith who provided great support and assisted with the computer graphics.

All the experiences that I gained over these three years has been the most unique and valuable in my life and it will never be a forgotten memory.



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Chapter 1

Introduction

Since the glass ionomer cements were first developed by Wilson and Kent in 1971, they have been commonly applied as luting cements, lining materials, restorative materials, core materials, and endodontic filling materials in dentistry. In another area, the cement is used as a biomaterial such as bone cement. However, their relatively inferior physical properties such as brittleness and low abrasion resistance (McCabe *et al.*, 1979; McLean, 1988; Forss *et al.*, 1991) have often been regarded as crucial disadvantages of these materials when compared with other restorative materials. Particularly in the early stages of development, there was not much information on the usage and characteristics of these materials to optimize their properties. Consequently the initial reputation of GICs was not favourable, and to some extent this impression has continued among some clinicians, in spite of the fact that many good and unique properties of GICs have become apparent. These unique properties were reviewed by Kao *et al.* (1996). They include:

- 1) Improved biocompatibility to dental tissues and chemical bonding to both enamel and dentine (Maldonado *et al.*, 1978; Coury *et al.*, 1982; Wilson *et al.*, 1983).
- 2) Anti-caries capability due to the release of fluoride ions into adjacent tooth structures (Hicks *et al.*, 1986; Silverstone, 1986; Purton and Rodda, 1988; Tyas, 1991; Griffin *et al.*, 1992; Hicks and Flaitz, 1992; Souto and Donly, 1994).
- 3) Inhibition of bacterial growth from sustained long-term fluoride release (DeSchepper *et al.*, 1989; Heys and Fitzgerald, 1991; Palenik *et al.*, 1992) and promotion of remineralization of unaffected inner dentine and demineralized enamel (Forsten, 1977).
- 4) Low coefficient of thermal expansion, similar to that of tooth structure (McLean and Gasser, 1985) which helps to minimize microleakage at the tooth-enamel interface (Gordon *et al.*, 1986; Craig, 1989).
- 5) Bonding to nonprecious metals and plastics (Hotz *et al.*, 1977).

Recently, there have been several novel developments in GICs aimed at overcoming the unfavourable physical properties. One new initiative has been the inclusion of a resin component into the glass ionomers to give what has been termed 'resin modified glass-ionomer cements' (RM GICs) (McLean *et al.*, 1994). These developments contributed to the popularity of GICs among a significant number of clinicians, because they were assumed to inherit two

very attractive characteristics that traditional GICs have. Firstly, GICs are the only dental materials currently available which show evidence of chemical adhesion to dentine (Tyas *et al.*, 1989). Secondly, GICs have been shown to give continuous fluoride release for a long period which can help the remineralization process in decayed tooth structures. There are numerous references to the bond strength and fluoride release of various GICs in the literature. However, to discuss them in detail is not within the scope of this study. Instead, two important additional properties (physical strength and wear resistance) have been investigated. In particular this study has focused on GICs as restorative filling materials rather than luting cements or lining cements. Also, the hybrid materials, so called 'polyacid-modified composite resins' (McLean *et al.*, 1994), which do not promote enough acid-base reaction were excluded from the discussion.

Chapter 2

Purposes of This Study

GICs and CRs are available as restorative materials, however, these two kinds of materials have different functions, properties and handling characteristics. Clinicians often face questions about which of the two common adhesive materials will give optimal results in the oral environment--- GICs or CRs? In many cases CRs show stronger physical properties and give more aesthetic results than GICs although the problems related to the polymerization shrinkage have not been overcome. On the other hand, GICs have advantages such as fluoride release and chemical bonding to tooth structure and thus GICs are the materials of choice in some situations. However, the poor physical properties are often described as a disadvantage of GICs. Could the new innovations such as an inclusion of a resin component to GICs improve their properties and expand the range of their clinical applications?

The purposes of this study were (1) assess the reliability of the shear punch test and establish experimental procedures; (2) investigate the unique properties of GICs and compare the shear punch strengths with CRs; (3) establish experimental procedures for the GIC wear study using the wear simulation machine; (4) assess the durability of three selected GICs as the restorative materials in terms of the wear resistance and strength.

Although one property does not explain all the clinical behaviours in the oral environment, the information obtained from this study is aimed to supply useful data to assist in the selection of appropriate materials.

Chapter 3

Literature Review

3.1 Development of GICs

The glass ionomer cements were invented in the Laboratory of the Government Chemist in England and developed by Wilson and Kent, (1963, 1971, 1972). GICs set by means of chemical gelation as a result of the reaction of an acid with a base (Wilson and McLean, 1988). These cements evolved from the dental silicate cements because of the general dissatisfaction with the clinical performance of the dental silicate cements. They were subject to clinical failures because of their high solubility in the oral environment and subsequent disintegration, and were not able to be further improved. In the same period, Smith (1968) first used polyacrylic acid in his zinc polycarboxylate cements, in what were later called polycarboxylate cements. Eventually, Kent *et al.* (1973,1979) found a glass that was high in fluoride (G-200), and finally after considerable research, Wilson and Kent developed the GICs. The name of this new cement was coined by Kent, and has been described as a hybrid of dental silicate cements and zincpolycarboxylate cements.

The cement was originally known as ASPA, an acronym for Alumino-Silicate Polyacrylic Acid. The first GICs lacked workability and hardened slowly. For these reasons, despite the many advantages that glass ionomers have to offer, the early version of the conventional cements were not extensively used as restorative materials. Reasons for the limited clinical acceptance of these cements were their relatively poor physical properties and aesthetics compared with other restorative materials such as composite resins. Since then, many improvements have been introduced to compensate for the physical properties and workability of the material, while maintaining the unique properties such as long term fluoride release and the ability to adhere to dentine.

Significant progress occurred when the so called 'dual-cured' glass ionomer cements were developed (Antonucci *et al.*, 1988; Mitra, 1989; Mathis and Ferracane, 1989; Minnesota Mining and Manufacturing Company, 1989; Wilson, 1989, 1990; Albers, 1990). These materials are curable by light activation, as well as a chemical reaction, and can thereby eliminate the long wait necessary for the setting of conventional cements. Manufacturers claim a number of advantages compared with conventional chemically cured GICs. However, there are many types of products on the market at present, and the setting reactions and properties

vary between products. This has led to some confusion in this rapidly developing area.

3.2 The definition of GICs

There is a world-wide non-commercial organization, the International Organization for Standardization (ISO), which establishes specific standards and defines standardized tests for many products. The assessment of dental materials is included as part of the ISO's responsibilities, and the standards are well accepted in the dental field.

ISO has presented the requirements and test methods for all types of water-based dental cements. In this standard glass ionomer cements are officially given the name "glass polyalkenoate cements". The term "glass ionomer cement" or "glass ionomer cements" is now used as a generic term to cover these cements. In this report, the term "glass ionomer cement (GIC)" or "glass ionomer cements (GICs)" is used to represent this generic term. The original GICs set by a simple acid-base reaction and are usually called "conventional GICs" or "chemically-cured GICs" or "auto-cured GICs" in the literature. In this report, the term 'conventional GICs' is used to describe this group of materials.

More recently, new variations of GICs which include a resin component have been introduced. In the early stage, they were often called "dual-cured", "light-cured" or "hybrid" GICs. Since then, the terminology has become more confusing. Mount (1994a) emphasized the problems of nomenclature that manufacturers and researchers use, and suggested the need for the clarity and unambiguous use of terms for GICs. For example, when one looks at the 73rd general session of the International Association of Dental Research (1994), there are a variety of terms in this new field, such as, "hybrid glass ionomer", "light cured glass ionomer", "dual-cured glass ionomer", "tri-cured glass ionomer", "methacrylate modified glass ionomer", "dark cured hybrid ionomer", "resin-reinforced glass ionomer", "resin ionomer", etc.

Manufacturers have produced further confusion by naming their products "compomers" and "ionoposites" to imply a link to GICs, and in fact they are actually composite resin with none of the beneficial properties which conventional GICs have (Mount 1994a). McLean *et al.* (1994) recognized the same problems and suggested the need to set the definitions and nomenclature for this field. They are described as follows:

- "Glass ionomer cements":

'A cement that consists of a basic glass and an acid polymer which sets by an acid-base reaction between these components'.

● "Acid-base reaction"

This must take place as part of the cement-forming process and not be delayed, for example, by the presence of excessive concentrations of organic components. This acid-base reaction is further defined to have 9 characteristics which include:

- Hard substance upon setting
- Low reaction exotherm
- No polymerization shrinkage
- No free monomer present
- Dimensional stability
- Filler-Matrix interaction
- Adhesion to enamel and dentine
- Fluoride release
- Early moisture sensitivity requiring protection (e.g., with varnish) immediately after placement

For hybrid materials that retain a significant acid-base reaction as part of their over-all curing process, McLean *et al.* (1994) suggested use of the term "resin-modified GICs" as the trivial name, and "resin modified glass-polyalkenoate cements" as the systematic name for use where more precise chemical nomenclature is appropriate, such as in the ISO standards. This term, "resin-modified glass ionomer" was originally used by Antonucci *et al.*(1988). Therefore, materials that do not set without light should not be called "resin-modified GICs", as the essential feature of these cements is that an acid-base reaction is critical for their setting, thus, they will set in the dark, albeit more slowly and yielding a material that is inferior to that obtained by photocuring .

It has been suggested that the other materials in which the correct ingredients are present in its components (i.e., acid decomposable glass, possibly some polymeric acid), but in insufficient amounts to promote an acid-base reaction in the dark, should be described as "polyacid-modified composite resins". Simonsen (1994) stated that these definitions are quite clear and useful in resolving the confusion in this field. In this report, the definition of "resin-modified GIC (RM GIC)" or "resin-modified GICs (RM GICs)" proposed by McLean *et al.*(1994) has been adopted.

3.3 Chemistry of GICs

3.3.1 Basic chemistry of GICs

Conventional glass ionomers combine the technology from silicates and zinc polyacrylate materials in order to incorporate the favorable characteristics of both these materials. The setting of glass ionomer cements has been characterized as an acid-base reaction between the polymeric carboxylic acid and the basic fluoroaluminosilicate glass (Wilson and McLean, 1988). Water plays an important role in this reaction by interacting with the polyacid to break the internal hydrogen bonding of the acidic carboxylic groups and make them more readily available to react with the glass. This acidic solution causes partial dissolution of the glass particles to elute or expose numerous ions, such as, fluoride ions and other ions, and this polyacid causes gelation (Figure 3.1).

There are three methods in which the polymeric acid can be mixed. It may be: 1) dissolved in water containing tartaric acid which is mixed with the glass powder, 2) freeze-dried polymer acid may be blended with the glass powder, which is mixed with an aqueous solution of tartaric acid, or 3) freeze-dried polymer acid and tartaric acid may be blended with the glass powder which is mixed with water. In each system, hydrated protons from the polymer acid attack the surface of the glass particles, releasing Al^{+3} and Ca^{+2} ions as well as fluoride droplets, which remain free and are not part of the matrix. The exact setting reaction is not clear, however, it has been suggested that the calcium polyacrylate chains form first, and then the aluminum chains follow immediately and strengthen the cross-linking effect. Wilson and Nicholson (1993) discussed the polyalkenoic cement setting reaction in detail, and described the following processes:

- 1) On mixing the cement paste, the calcium aluminosilicate glass is attacked by hydrogen ions from the polyalkenoic acid and decomposes with liberation of metal ions (aluminum and calcium), fluoride and silicic acid (which later condenses to form a silica gel).
- 2) As the pH of the aqueous phase rises, the polyalkenoic acid ionizes and most probably creates an electrostatic field which aids the migration of liberated cations into the aqueous phase.
- 3) As the polyalkenoic acid ionizes, polymer chains unwind as the negative charge on them increases and the viscosity of the cement paste increases. The concentration of cations increases until they condense on the polyacid chain. Resolution occurs and insoluble salts precipitate, first as a sol which then converts to a gel. This represents the initial set.
- 4) After gelation or initial set, the cement continues to harden as cations are increasingly

bound to the polyanion chain and hydration reactions continue. Recent evidence suggests that a siliceous hydrogel may be formed in the matrix.

Fluoride clearly has a considerable effect on the reaction, as it is believed to form strong soluble complexes with aluminum such as AlF^{+2} and AlF_2^+ (Connick and Poulsen, 1957; Akitt *et al.*, 1971). Originally, it was supposed that this attack occurred only at the surface layer of the glass particles (Barry *et al.*, 1979), but a later study by Ellison and Warrens (1987) suggested that attack occurred throughout the glass particles.

A similar chelation reaction can take place between the setting ionomer mix and calcium on the surface of the tooth structure, resulting in an adhesive bond.

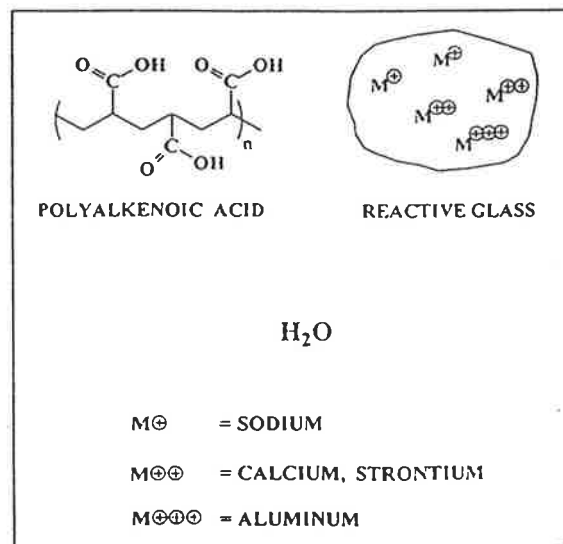


Figure 3.1

Classical glass ionomer chemistry involving three major elements: polyalkenoic acid, reactive glass and water (Hammesfahr 1994)

3.3.2 The role of water

Water plays an integral role in the setting and performance of glass ionomer cements. Wilson and Nicholson (1993) showed three possible roles of water. Firstly, water aids in the dissolution, release and mobility of the ionic species. This is vital to the formation of the poly-carboxylate complex which results in the setting of the glass ionomer cements, as well as in the sustained fluoride release from these cements. Secondly, it is one of the components of the set cement, and becomes incorporated into the cement as it hardens. Thirdly, water may react as a plasticizer, and it affects rigidity, dimensional stability and diffusion coefficients within the bulk.

Although, water is the basic component of GICs, water contamination works as an inhibiting factor in the initial stage of the setting reaction. Water weakens the bonds between molecules, worsening the subsequent properties of the hardened cements, for example, showing white spots, wear and tear, craze lines, fractures, discoloration and staining (Saito, 1993). Early protection against water is overcome clinically, to some extent, by using some sort of protection such as varnish to seal the cement during its early life (Wilson and McLean, 1988). However, Earl *et al.* (1985) stated that this does not give perfect results, and as yet there is no ideal barrier material for this purpose.

It is clinically significant that the degree of water sensitivity is greater and extends into deeper areas of the cement earlier in the setting reaction, but decreases as the setting reaction proceeds, and once a certain stage is passed, water sensitivity disappears (Saito, 1993). These two different stages of the water sensitivity of GICs have been explained as 'loosely bound' and 'tightly bound' water in the set cement (Wilson and McLean, 1988). GICs consist of approximately 24% water, which can be divided into approximately 5% 'loose water', which easily evaporates by dehydration, and 18-28% 'tight water' which is bound to the matrix structure (Prosser and Wilson, 1979). The greater proportion of the water in aged cements has become 'tight water', therefore, the cements become stable in both water and air (Saito, 1993).

3.3.3 Improvement of GICs

There have been many attempts to improve the physical properties of GICs based on this knowledge of their chemical reaction. Wilson and McLean (1988) described the factors that effect the physical properties of GIC as:

- 1) Powder/ liquid ratio (P/L)
- 2) Time to mature

- 3) Water balance
- 4) Porosity
- 5) Molecular weight of the liquid and the difference of the glass powder

One innovation has been inclusion of tartaric acid into the formula to accelerate the setting reaction (Wilson and Crisp, 1975, 1976, 1980). It shortens the setting time, increases the hardening rate and increases the strength without decreasing working time (Crisp *et al.*, 1979). Moreover, this innovation enabled the usage of lower fluoride content glasses which were more translucent than high fluoride content glasses, and thus aesthetics of the restoration could be improved.

With an increase of polyacid concentration, the strength of the cements was reported to increase almost linearly (Crisp *et al.*, 1977a). However, increase in concentration of the polyacid increased solution viscosity quite sharply causing stiff mixing and loss of working time.

The inclusion of the higher molecular weight polyalkenoic acid could also contribute to the improved physical property of the cements. Strength, fracture toughness, resistance to erosion and wear were all improved as the molecular weight of the polyacid was increased (Wilson *et al.*, 1977; Wilson *et al.*, 1989). However, as the molecular weight increased, the viscosity of liquid also increased and therefore clinical handling was difficult and necessitated a reduction in P/L. This problem was later solved by drying the polyalkenoic acid and blending it with glass powder and using water or diluted tartaric acid as the liquid. In this type of GICs, water could be used to initiate cement formation with the benefit of lowering the stiffness of the mix (McLean *et al.*, 1984; Prosser *et al.*, 1984). This permitted the use of polyalkenoic acid of higher molecular weight. Atkinson and Pearson (1985) studied the water/powder type GIC, and reported considerable improvements in the properties. The addition of metal fluorides to formulations had been found to accelerate cement formation and increase the strength of set cements (Crisp *et al.*, 1980a). The effect was enhanced by the presence of tartaric acid.

Inclusion of amalgam alloy was introduced to improve the physical properties of conventional GICs. They are divided into several kinds as follows:

- 1) Addition of amalgam alloy particles (Simmons, 1983)
- 2) Addition of stainless steel particles (Kerby and Bleihdder, 1991)
- 3) Sintering of silver metal with the glass powder (McLean and Gasser, 1985)

Ketac-Silver (ESPE, Seefeld, Germany) is a commercially available example of the third type in which conventional fluoroaluminosilicate glass is reinforced by sintering with metal. These products were sometimes called "cermets". According to the study by Walls *et al.* (1987), Ketac-Silver exhibited an increase in compressive strength and compressive fatigue limit, however, flexural strength was significantly lower than traditional GICs. Other researchers have reported similar properties (Goldman, 1985; Lloyd and Adamson, 1987; Lloyd and Butchart, 1990). In addition, the adhesion to tooth structure of the cermet was found to be inferior to conventional GICs (Thornton *et al.*, 1986) and leaching of greyish metallic particles into dentinal tubules has been reported as a significant aesthetic problem (Sarkar *et al.*, 1988). Significant decrease of fluoride release from cermet and related anticariogenicity was reported (Swift, 1989). Therefore, the presence of the silver on the surface of the particles allowed moderate improvement in abrasion resistance, but the over-all properties of GICs were not significantly improved by these modifications.

It was claimed that the flexural strength and compressive strength of the cements which incorporated amalgam alloy particles into the cement powder (such as Miracle Mix, GC Corporation, Tokyo, Japan) have been improved (Simmons, 1983). However, it did not increase the compressive strength (Beyls *et al.*, 1991; Williams *et al.*, 1992). Consequently, the overall physical properties of this type of cements did not fundamentally change, since there was no union between the metal and cement (Mount 1994b).

3.3.4 Inclusion of a resin component

One of the most recent developments has been the advent of GICs with the inclusion of a resin component. These materials are curable by light activation and can thereby eliminate the long wait necessary for the setting of conventional GICs. However, it is important to realize that the constituents of the various types of GICs currently available are not the same. Some are merely a variation of composite resin, which do not have an acid-base reaction at all in its system, whereas others still keep the original favorable properties of GICs (McLean *et al.*, 1994).

It is reported that the inclusion of a resin component into GICs has shown improvement of their physical properties and some data on the mechanical properties of RM GICs have been published (Antonucci *et al.*, 1988; Mathis and Ferracane, 1989; Mitra, 1989, 1991; Albers 1990; Katsuyama *et al.* 1993; Tosaki and Hirota, 1994). Beside this improvement, RM GICs have a longer working time because HEMA (2-hydroxyethylmethacrylate) slows the acid-base reaction, yet sets sharply once the polymerization reaction is initiated by light. The

manufacturers claim that RM GICs are also resistant to early contamination by water because of the formation of an organic matrix, therefore, it is claimed that no protection by varnish is required. While these characteristics in handling are attractive for clinicians, the test results of these new materials should be carefully interpreted. Due to the relatively recent introduction of these products, much of the data comes from patents and company reports, so that it is impossible to draw firm conclusions from this information alone (Wilson and Nicholson 1993). For example, these authors commented that RM GICs appear to have 'rubbery' characteristics in the early stage of setting, and concluded that this rubberiness will disappear as the cements age and the acid-base reaction is completed. Nicholson *et al.* (1992) reported that long term storage in water caused a decline in the physical properties of RM GIC liners, but this is not supported by other published studies (Lewis *et al.*, 1992; Mitra 1991).

There are several different approaches to creating a new hybrid material from ionomer and polymer chemistry, but they are able to be simplified into modifications of the powder, modifications of the liquid portion or modifications of both. Burgess *et al.* (1994) have described a continuum between traditional composites and conventional GICs that includes the modified materials at various 'intervals'. As previously described, there is some confusion in the definition of recently developed GICs with a resin component. The characteristics of some materials should be more accurately described as "fluoride-releasing resins" or "polyacid-modified composites" and distinguished from "resin-modified GICs" which demonstrate the proper acid-base reaction (Burgess *et al.*, 1994; McLean *et al.*, 1994).

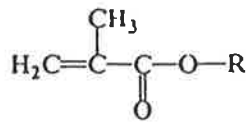
Hammesfahr (1994) discussed these differences and stated that a number of current materials contain elements of both glass ionomers and composites, and the distinction between the two classes of materials is becoming blurred. The author summarized them as follows and they are discussed in more detail in the following sections:

- 1) Polymerizable monomer/ Prepolymer substitution for polyalkenoic acid
- 2) Polymerizable monomer/ Prepolymer addition to polyalkenoic acid
- 3) Polymerizable polyalkenoic acid
- 4) Acid monomer

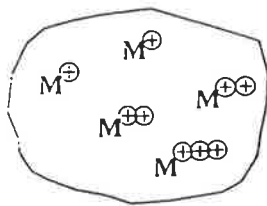
Of these four categories, the materials listed in 2) and 3) may correspond to the "RM GICs" defined by McLean *et al.* (1994).

1) *Polymerizable monomer/ Prepolymer substitution for polyalkenoic acid*

The materials with this modification are more like traditional composite resins because of the lack of two important ingredients of acid-base reaction, namely, the acid and water. An example of this type of material would result from adding glass to a known polymerizable methacrylate system, such as Bis-GMA, in the absence of water to form a composite material (Figure 3.2). Obviously, few of the advantageous properties of the ionomer chemistry would survive using this modification. Examples of commercially available products of this type include GeriStore™ (DenMat), Resinomer™ (Bisco) and Ionoseal™ (Voco).



MONOMER, PREPOLYMER



REACTIVE GLASS

NO WATER

NO ACID

Figure 3.2

Polymerizable monomer/ prepolymer substitution for poly-alkenoic acid in hybrid resinomer systems (Hammesfahr 1994)

2) *Polymerizable monomer/ Prepolymer addition to polyalkenoic Acid*

This system blends the polyacrylate or polyalkenoic liquid with monomers that can be polymerized, usually with visible light (Figure 3.3). The polyacid, monomers, and water can be homogeneous, typically by use of a hydrophilic monomer, such as 2-hydroxyethylmethacrylate (HEMA). Although the reaction between the glass filler and the polyacid is not exactly the same as the traditional GICs, an acid-base reaction remains in place and the reaction can incorporate two parts, namely, self curing and free radical polymerization. Thus, these systems are referred to as "dual-cure" or "tri-cure" materials. Examples of commercially available products in this approach include VariGlass™ (Caulk/ Dentsply), Photac-Fil™ (Espe-Premier) and Fuji II LC™ (GC Corporation).

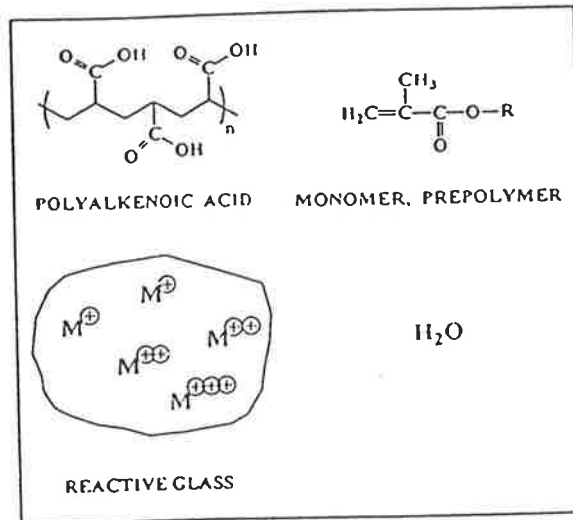


Figure 3.3

Major elements in the polymerizable monomer prepolymer addition to the polyalkenoic acid of traditional glass ionomer chemistry (Hammesfahr 1994)

3) Polymerizable Polyalkenoic Acid

The formation of this type of product is through the pendant carboxylic acid of the polyacid attacking polymerizable side groups (Figure 3.4). The polyacid backbone remains essentially intact with additional polymerizable side chains as part of the structure, and thus the traditional glass ionomer system remains. Examples of commercially available products in this approach include Vitrebond™ (3M) and Vitremer™ (3M).

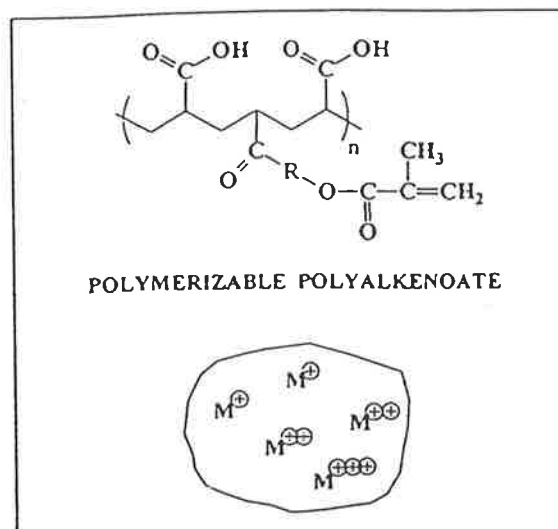


Figure 3.4

The polyalkenoic acid molecule is reacted with polymerizable materials to create a polymerizable polyalkenoate that may react with the reactive glass in the presence of water (Hammesfahr 1994)

4) Acid monomer

Another approach for modifying GICs is the substitution of acidic polymerizable monomers for the polyalkenoic acid polymer. In this system, a relatively small molecule containing both acidic and polymerizable functionalities, replaces the large, high molecular weight polyacid (Figure 3. 5). Dyract™ (Dentsply) is an example of a commercially available product in this category.

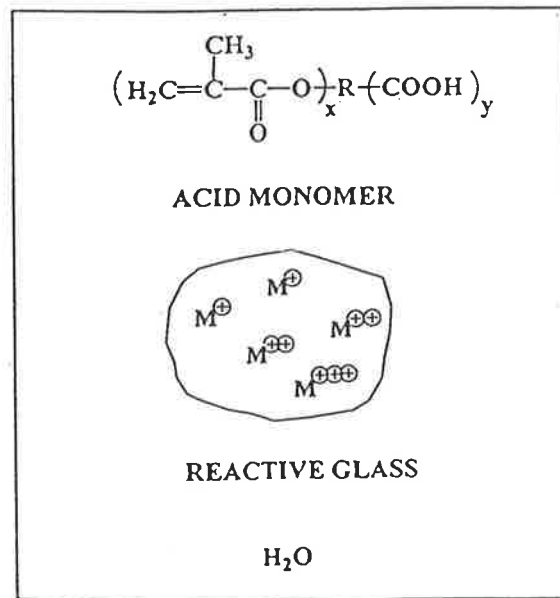


Figure 3. 5

Acid monomers replace the polyalkenoic acid while the other elements of traditional glass ionomers (the reactive glass and water) remain (Hammesfahr 1994)

3.3.5 Maturing effect of GICs and physical properties

It is reported that the setting reaction of GICs continues for months after the cement has been formed (Crisp and Wilson, 1974). Crisp *et al.* (1976a) reported a continuous increase in the compressive strength of the original ASPA glass ionomer cement over time. This change of the quality of GICs over time is sometimes described as a "maturation" or "aging" effect.

There are several factors which influence the early physical strength of the cements. One is the chemical composition and microstructure of the glass (Prosser *et al.*, 1986). Powder/liquid ratio (Crisp *et al.*, 1976b; Prosser *et al.*, 1986) and the nature, concentration and molecular weight of the polycarboxylic acid (Crisp *et al.*, 1977a, 1977b; Wilson *et al.*, 1989) also influence the early physical strength. Wilson *et al.* (1979) suggested that increased physical strength of GICs over time was due to the increase in the hydration of the metal-carboxylate links.

On the other hand, decreased physical strengths of GICs after long-term storage in an aqueous

environment was reported (Pearson and Atkinson, 1991; Williams and Billington, 1991; Soltesz and Leupolz, 1992). This could happen due to the water absorption of these materials. An *in vitro* study by Cattani-Lorente *et al.* (1994) showed most conventional GICs stored in water over a long-term maintained constant strength.

There is some controversy regarding the effect of water on RM GICs in which the HEMA component is supposed to be more hydrophylic than conventional GICs. Reports of long-term studies on the more recently available RM GICs are quite limited (Mitra and Kedrowski, 1994). However, Nicholson *et al.* (1992) studied two RM GIC liners stored in water over 90 days, and found the compressive strength was not affected. Also, Mitra (1991) showed no significant effect on the strength on resin-modified GIC base liner/base stored in water over 7 months. In a clinical study, Croll (1991) found that RM GICs performed well after 2 years. These data appear to support the hypothesis that RM GICs may have long-term physical integrity comparable to that of conventional GICs (Mitra, 1994).

3.4 Specifications and standardized testing

Since about 1920, specifications for dental materials have been developed. They were originally developed by professional associations, such as the American Dental Association (ADA), and later by national specialized bodies (e.g., American National Standards Institution, ANSI; British Standards Institution, BSI; Deutsches Institute Fur Normung, DIN; Association Franchise de Normalisation, AFNOR; Standard Association of Australia, etc.) and the international specialized body (International Organization for Standardization, ISO). ISO is based in Geneva, and includes representatives of national organizations, and is developing its own standards.

The main purpose of the specifications is to provide the community with an undisputed tool for evaluating the aptitude of a material or device for its intended use (Meyer, 1994). Although the initial purpose of the specifications is clear, these are interpreted differently by the people involved in this process. For a manufacturer, the standards are basically quality controls. For a user of the product, specifications are considered as a safeguard. However, some significant characteristics of materials, such as long term degradation or biocompatibility may not have been fully addressed in the relevant specifications, consequently, this may give unfounded confidence in their use. Patients, on the other hand, are less likely to know of the existence of specifications, but once they know of their existence, they may consider the specifications as a guarantee of the long term service of dental materials. In fact, testing for long term durability is not often included in these specifications.

For researchers, although specifications may sometimes provide convenient methods for testing products, they are frequently so basic that performances cannot be determined from them, and more advanced techniques have to be selected or developed. Thus, in spite of the necessity for specifications, the current standards have 'not yet reached the level of completeness and efficacy' (Meyer 1994). For example, ISO 4049-1988 ('Resin based filling materials') does not refer to wear or/and fatigue testing, there is no description about filler content and there is no reference to the posterior composite filling materials. Obviously, one reason for this shortfall is that specifications are usually developed sometime after the materials are marketed. Especially in the last decade, progress in the development of dental materials has been so rapid that specifications often may not fit with the latest materials when they are issued.

While specifications in dentistry have for a long time been considered as a set of rigid rules for mechanical properties, the recently developed ISO standard for 'Biological evaluation of medical devices' included dental materials as a medical device (ISO/ TC 10993 Biological evaluation of medical devices). Meyer (1994) stated that this inclusion of dental materials in this standard is favorable as it will enhance the development of dental materials' biosafety aspect. The same author believed that researchers should propose to the standardization bodies better testing procedures which reflect more realistic behaviour of dental materials in the oral environment.

• GIC specifications

Since 1991, ISO has specified the requirements and test methods for all types of water-based dental cements in one document (9917: 1991, 'Dental water-based cements'). Several ISO standards that were previously published for the individual cements described below were therefore, withdrawn:

- ISO 1565: 1978, Dental silicate cement (hand mixed)
- ISO 1566: 1978, Dental zinc phosphate cement
- ISO 3824: 1984, Dental silicophosphate cement (hand-mixed)
- ISO 4104: 1984 Dental zinc polycarboxylate cements
- ISO 7489: 1986 Dental glass polyalkenoate cements

Current requirements consist of 1) film thickness (for luting cements only), 2) setting time, 3) compressive strength, 4) acid erosion, 5) opacity (for restorative cements only), 6) acid-soluble arsenic and lead contents. In the same document, it is suggested that reference should be made

to ISO/TR 7405: 1984, Biological evaluation of dental materials or any more recent edition regarding the requirement of biological or toxicological hazards. However, this is one of the examples of a standard lacking any reference to long-term assessments, and new hybrid materials such as RM GICs are not easily categorized for assessment according to these standards. Moreover, it is mainly focused on mechanical properties and the assessment of the biocompatible factors are not included. Consequently, there are no requirements for prescribing the microleakage, bond strength or wear characteristics which are all clinically important.

Recently, a new ISO standard (draft) for 'Light Activated-Water-Based Cements' was issued (1996 ISO DIS 9917 Part 2, Water based cements light activated cements). This document describes the hybrid GIC products which can be described as 'water-based and set by multiple reactions which include an acid-base reaction and polymerization'. In other words, this new standard is based on the point of the applications rather than on chemical typing (McCabe, 1996).

3.5 Assessment of the physical strength

3.5.1 Introduction

Mechanical properties are an important test because most restorative materials must withstand forces during mastication. However, there is no single mechanical property which can give an overall indication of the clinical performance of a material and it is essential to understand the limitations of these tests.

There are many ways to test the mechanical properties of dental materials. It should be noted that the properties of dental materials vary and range from very hard materials such as metals, elastic materials such as impression materials to very brittle materials such as gypsum, ceramics, some impression materials and cements. Thus, each test is restricted to certain materials, and it is not possible to adapt one single testing procedure to measure all of these different materials as some tests when applied to certain materials are not likely to give clinically relevant information. It should also be recognized that these tests are often used as industrial quality control tools, and are not directly related to the exact prediction of the clinical behaviour of these materials.

There are several test methods to estimate the mechanical properties of materials. Strength is formally defined as the force experienced by a material at the point where fracture occurs (Gilliam 1969). The most common ways to test strength in dentistry are:

a) Tensile strength test

- b) Compressive strength test
- c) Shear strength test
- d) Flexural strength test (or Transverse strength test, Three point bending test)

In addition to these traditional strength tests, two alternative tests which are especially useful to assess physical properties of GICs were also reviewed in this section:

- e) Fracture toughness test
- f) Shear punch test

Generally, if plastic flow as a mean of failure is excluded, the materials can only fracture in one of two ways:

- by the pulling apart of planes of atoms, (i.e. Tensile failure)
- by the slippage of planes of atoms (i.e. Shear failure)

Therefore, the results of these tests should consider a mixture of these two failure types. Generally, strength is determined by applying forces uniaxially using an apparatus consisting of jaws which move either together or apart in a controlled manner. A chart recorder is employed to give a permanent record of the results obtained, so that the force at fracture can be determined (Wilson and Nicholson, 1993). The results of these tests depend on the size and geometry of the specimens, the nature of the material tested and the rate of loading, thus, the values should be interpreted as a means of comparison between similar materials (Darvell, 1990).

3.5.2 Common testing methods of the physical strength

3.5.2.1 Tensile strength test

Tensile strength tests were developed to test ductile materials, such as metals, alloys, and some plastic substances. The tensile strength is generally determined by subjecting a rod, wire or dumbbell-shaped specimen to tensile loading. Brittle materials like GICs which tend to rupture suddenly with minimal deformation, are usually difficult to test in this traditional method. Because mounting the specimen often involves gripping the materials, it creates concentrated stress in certain areas, where premature fractures may occur. Generally, there has been large variability in tensile data on brittle materials (Craig 1993), and such tests need sufficient samples to get enough data for statistical analysis, so the method is very time consuming. An alternative method for testing brittle materials is called the diametral compression test for

tension or Brazilian test. In this method, the compressive load is placed on the diameter of a short cylindrical specimen (Figure 3.6). This method involves relatively easy specimen preparations and operations especially when brittle materials are tested. The tensile stress is directly proportional to the load applied through compression and is measured using the following formula:

$$\text{Tensile stress} = \frac{2P}{\pi DT}$$

P = Load

D = Diameter of the specimen

T = Thickness of the specimen

Although this method is widely used in assessing the mechanical strength of dental materials, Wilson and Nicholson (1993) stated that interpretation of results of this test was uncertain because of the complexities in the mode of failure. Williams *et al.* (1992) also stated that the diametral compressive test was not suitable for GICs. Minor imperfections, such as voids, in the specimen can lead to localized stress concentrations which affect the magnitude of the results.

3.5.2.2 Compressive strength test

This test is more clinically important when compared to the tensile strength test, as the failure of restorations is often related to the mastication process. Usually the specimens are a cylinder shape and a uniaxial force is applied. ISO (9917; Dental water based cements, 1991) specifies the compression test as the standard test for dental water based cements. It recommends the specimen size as 4.0 mm diameter and 6.0 mm in height.

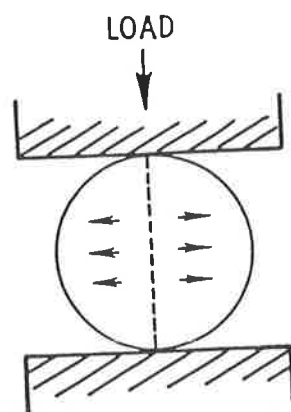


Figure 3.6

Diametral tensile test. A compression load is applied diametrically to a cylindrical specimen. The arrows indicate the direction of the tensile stress. (Phillips 1991)

3.5.3 Other testing methods of physical strength

3.5.3.1 Fracture toughness test

Fracture toughness test is discussed as an alternative method for evaluation of the mechanical property of the cements. In previous tests, such as compressive, tensile and flexural tests, failure is catastrophic because there is no suitable flaw for crack propagation. In fracture toughness tests, the specimens generally have artificial minute flaws, so that stress is concentrated at these flaws and enhanced. Thus, materials under this condition do not reach their theoretical strength when fractures occur.

Douglas and Lin (1994) recommended the Chevron-notched short-rod fracture toughness test as the preferred test for GICs . This method was first introduced by Barker (1977) and its geometry of the short rod specimen is illustrated in Figure 3.7 and Fig 3.8. This technique was originally developed in the field of engineering for testing strain fracture. The propagation of the crack starts at the 'V' shaped tip (Figure 3.7).

Douglas and Lin (1994) emphasized that fracture toughness is more clinically relevant than compression strength since the clinical catastrophic failure of the restoration occurs when fracture propagation occurs. This explains why materials often fail well below their referenced average stress-based strength. Wilson and Nicholson (1993) discussed a double torsion fracture toughness test which could be beneficial for the estimation of GICs. In this method, specimens take the form of rectangular plates with a sharp groove cut down the center to eliminate crack shape corrections. An initiating notch is cut into one end of each specimen (Hill and Wilson 1988), and after that, the specimens are tested on two parallel rollers, resulting in the crack being propagated along the groove. More details of this test are discussed in their paper.

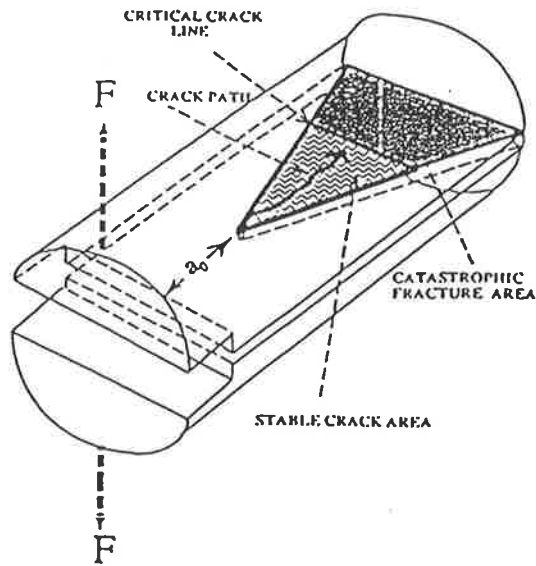


Figure 3.7
Chevron-notched short rod specimen configuration (Douglas and Lin, 1994)

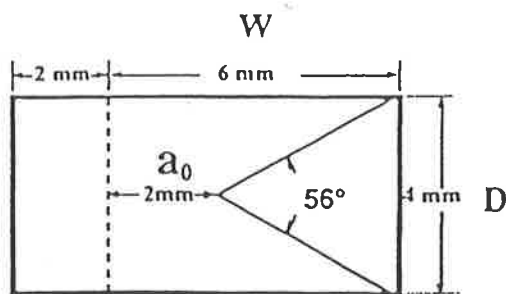


Figure 3.8
Geometric dimensions of the chevron notch (Douglas and Lin, 1994)

3.5.3.2 Shear punch test

The work of Mount *et al.* (1994) described a shear punch test as a preferred test for light cured materials. This method was first introduced by Roydhouse (1969, 1970) for gauging a dental material's strength by punching out a small disc (3 mm in diameter) from a thin sheet of the material (1.0 mm thick). Later, this method was further defined by Smith and Cooper (1971).

Mount *et al.* (1994) examined several light cured materials, including RM GICs with this method and concluded that this test was less technique sensitive, had a small standard deviation and was an adequate test for light cured materials due to the specimen size. At present, ISO (9917, Dental water based cement, 1991) specifies cylindrical specimens (4.0 mm in diameter, 6.0 mm in height) for compression testing of dental cements, but for light cured materials, the authors believed the size of the specimen was not suitable as the cylinder must be built in layers due to the depth of cure. Anstice *et al.* (1992) examined the effect of building specimens using the layer technique on the compressive strength of resin-modified GICs. They prepared layered and unlayered specimens of two different sizes, and found the compressive strength values did not change with different specimen sizes, but did with layered specimens, and concluded that the use of layered specimens was not a valid variation of the compressive strength test. Therefore, this method could be an alternative from the current standardized test to control the quality of the resin-modified GICs. Moreover, this method is also useful for other light cured CRs. ISO (4049, Resin based filling materials, 1988) specifies the flexural strength to estimate the strength of resin-based filling materials as a standard test, but, again, manufacturing long thin specimens which are set to the size of (25 ± 2) mm x (2 ± 0.1) mm x (2 ± 0.1) mm is difficult. This shear punch test seems to provide a more simple and useful method for investigating the properties of light-cured materials.

3.5.4 Conclusion

The information regarding the physical strength of GICs is particularly important for their clinical usage as restorative materials. GICs are generally known as brittle materials and the developments of RM GICs which have been reported to display superior physical properties to conventional GICs may be very beneficial in expanding their application *in vivo*. Also, improvement in physical properties of GICs may mean the improvement in the bond strengths of materials, because the bonding failure of GICs and tooth structure usually occurs with cohesive failure within GICs (Mount, 1991).

3.6. Assessment of material wear

3.6.1 Introduction

There are numerous *in vitro* and *in vivo* studies of material wear in the dental literature. Assessing dental wear is very challenging, because of its multicausative nature. Although *in vivo* wear studies are of paramount importance, they are difficult, time-consuming, expensive, and involve variables which significantly differ between both individuals and populations (Harrison, 1976; Abell *et al.*, 1983). Although *in vitro* studies do not reproduce the oral environment, they simulate certain oral conditions and allow for the control of a range of variables. However, it is often difficult to interpret differences between data obtained from *in vitro* and *in vivo* wear studies (McCabe and Smith, 1981).

3.6.2 In vivo wear assessments

3.6.2.1 Introduction

In vivo assessments of material wear generally involve one of two methods. The first method involves visual assessment by direct observation, and is often used in epidemiological studies where large sample sizes are required. However, limited access into an oral cavity, the presence of saliva etc. often makes this technique difficult. The second method is the use of replica techniques which overcome some of the problems arising from the first method. Teeth and restorations can be observed extra-orally so that more detailed information can be recorded. Different indices have been developed to assess tooth wear, but the value of such systems is limited as they are scored subjectively (Dahl *et al.*, 1993). Vrijhoef *et al.* (1985) reviewed the development of various methods for assessing material wear (Table 3.1), and stated that more sophisticated methods, though more accurate, are not surprisingly more expensive and time consuming.

3.6.2.2 Indices based on clinical observation

The rating systems called the 'USPHS evaluation systems' were developed by Ryge and Cvar (1971). Using this method, wear is quantified by a subjective evaluation of the anatomic form. The disadvantage of such systems is the subjectivity of the process which can lead to a reduction of the discriminating power (Vrijhoef *et al.*, 1985; Roulet, 1987; Dahl *et al.*, 1993). Leinfelder *et al.* (1989) proposed using a series of templates which allowed the quantification of the vertical substance loss of dental materials in an attempt to improve this type of evaluation. They reported that this reduced errors to approximately 20-30 μ m. The main criticism of indices is the fact that the clinical grading of wear is often not sensitive enough to quantify minor wear found in modern industrialized populations (Nystrom *et al.*, 1990).

Table 3.1

The observation methods for recording the loss of substance of dental restorations in the literature (modified from Vrijhoef *et al.*, 1985)

Authors	Methods
Ryge and Cvar (1971)	USPHS-criteria
Kusy and Leinfelder (1977)	SEM-pictures
Handelman <i>et al.</i> (1978)	Weight of material loss
Mettler <i>et al.</i> (1978)	3D Microscopy
Atkinson <i>et al.</i> (1982)	Laser optical counterering
Lutz and Mormann (1982)	Profilometer
DeLong and Douglas (1983a,b)	Extensometer
Roulet <i>et al.</i> (1983)	Precision occlusal mapping

3.6.2.3 Replica techniques

A common method for detecting detailed material wear and/or tooth wear involves what are described as 'replica techniques'. This indirect method provides more information about the mechanisms of wear than clinical visual assessment, and longitudinal records can be permanently kept and compared at a later stage (Hirt *et al.*, 1984). Impressions using silicone materials are taken and positive epoxy resin casts are subsequently made (Kusy and Leinfelder, 1977; Leinfelder, *et al.*, 1980; Roulet, 1987; Bailey *et al.*, 1988). Alternatively, copper-plated impressions can be cast into gypsum stone (Lambrechts *et al.*, 1982). This method, accompanied by the development of sophisticated computerized measuring systems, enabled a more detailed qualitative and quantitative analyses of wear to be made.

These techniques have been used to measure vertical loss of material from occlusal surfaces with a reported precision of approximately 20 μ m (Handelman *et al.*, 1978; Vrijhoef *et al.*, 1985). However, the disadvantage of these methods is the possible distortion of replica models due to curing shrinkage of resins (Lambrechts *et al.*, 1982). Ekfeldt *et al.* (1985) studied several combinations of epoxy resins and different impression materials and concluded that one silicone impression material (President™, Coltene) combined with one of the epoxy resin materials (Metapox™, Ivoclar) seemed to be the easiest to handle and give the best model.

3.6.2.4 Measuring wear using replica techniques

- *Profilometer technique*

Measurement of wear is possible by using a modified surface roughness tester or by a profilometer that traces a microscopic profile of the occlusal surfaces on the replica between two reference points (Lutz and Mormann, 1982; Harrison *et al.*, 1984; Hirt *et al.*, 1984). However, because this measurement is based on the profile record between two reference points, maximum wear at occlusal contacts might not be determined (Sulong and Aziz, 1990), and so estimating loss based only on localized sites cannot be used for accurate measurement of volume loss due to the two dimensional nature of the record (Harrison, 1985).

- *Stereometric technique*

Stereometric techniques enable contour plots to be generated by stereo-photographs. Originally, this method was used to monitor changes in the residual alveolar ridge (Adams and Wilding, 1985). The problem with this method is that it is technically difficult to obtain stereo-paired photographs, and obtain results in an analog form.

- *3D reflex microscope*

The reflex microscope permits direct measurements to be made in three dimensions without photography (Mettler *et al.*, 1978; Scott, 1981). This optical measuring device encodes for each axis, and data is transformed into digital values of the X and Y co-ordinates. The height of the object is able to be recorded by focusing a light spot onto a ghost image of the object. These data are stored on computer file, and software programs are available to draw a contour or a computer model of the object, or to plot and calculate areas and volumes from the stored data (McDowell *et al.*, 1988).

Adams and Wilding (1988) evaluated this computerized reflex microscope method using lead casts which were experimentally worn, and then compared it to a gravimetric method as a control. They found that the mean difference between these methods was 10 μ m. Pintando *et al.* (1991) used this system for their 30 month clinical study of resin white sealant which showed a volume loss of 0.43 mm³. However, these kinds of sophisticated techniques are often time consuming, and technically sensitive, thus, their usefulness in routine clinical situations is questionable (Vrijhoef *et al.*, 1985; Johansson *et al.*, 1993).

- *Other techniques*

The other measuring methods which can be made from replica techniques are by using laser

holographic (Moire) techniques (Atkinson *et al.*, 1982; Williams *et al.*, 1983), laser dual source contouring, and three-dimensional measuring techniques (Lambrechts *et al.*, 1984; Roulet, 1987; McDowell *et al.*, 1988). Some researchers have used SEM picture techniques to examine enamel wear qualitatively (Ekfeldt and Oilo, 1988, 1990).

3.6.2.5 Other methods to measure *in vivo* wear

The use of removable apparatus may be an alternative way to measure wear *in vivo*. For example, composite resin restorations placed in denture teeth have been used to investigate occlusal contact wear (Mitchem and Gronas, 1985). Ekfeldt and Oilo (1988) used a fixed bridge with two removable contralateral segments in their *in vivo* study, while Lindquist *et al.* (1995) assessed clinical wear of denture teeth. However, little research has been done using this technique as it is very costly and time consuming.

3.6.3 In vitro wear assessments

3.6.3.1 Measuring *in vitro* wear

There are a number of ways to assess *in vitro* material wear. One of them involves replica techniques. As previously described, profilometer and 3D reflex microscope, for example, can be used in the same manner as that *in vivo*. Another method which uses volumetric analysis based on calculated weight loss can be only used *in vitro*. Further method is using radioisotope techniques (Moores *et al.*, 1983; Harrison, 1985), measuring radiation in a slurry of irradiated material which was collected during wear process.

3.6.3.2 Problems of *in vitro* wear studies

Often clinical wear studies are difficult, time-consuming, expensive and involve complex factors such as variation of personal habits etc. (Harrison, 1976; Abell *et al.*, 1983). Therefore, most studies are conducted *in vitro*, and *in vivo* wear studies are less frequent (Ekfeldt, 1989). Especially the introduction of posterior composite resins has initiated a large increase in wear testing research and various *in vitro* wear testing machines and methods have been developed. (Ekfeldt, 1989). Table 3.2 summarizes some results of the *in vitro* wear studies.

However, it is often difficult to get a good correlation between the findings of *in vitro* and *in vivo* wear studies because it is very difficult to simulate the complex oral environment (McCabe and Smith, 1981). Roulet (1987) stated *in vitro* wear testing as 'one of the most challenging subjects in dental materials'. In addition, as there is no standard tests of wear study, researchers have adapted various methods to assess material wear. Moreover, many studies were carried out by using abrasive discs or papers and no tooth substance were involved. Some

studies involved tooth structure, these kinds of studies were less frequent in the literature (Heath and Wilson, 1977; Forss *et al.*, 1991). Harrison (1984) stated that many abrasion testing apparatuses do not reflect the situation *in vivo*, and the loads, speeds, and abrasives used are often more severe than would be encountered *in vivo*.

The other point is that the wear mechanisms are interpreted by researchers in various ways. For instance, Lutz *et al.* (1984) used a two body abrasion system, and De Gee *et al.* (1986) conducted studies involving three body abrasion to assess composite resin wear. Consequently, the results within the *in vitro* studies are often reported controversially. For example, some studies reported that cermets had shown less wear than conventional GICs (Moore *et al.*, 1985; McKinney *et al.*, 1988), while similar or more wear resistance of cermets has been reported in other studies (Walls *et al.*, 1987; Forss *et al.*, 1991)

Table 3.2
Reported *in vitro* wear studies of various restorative materials

Authors	Wear rate ranking Ratio*	Wear
O'Neal and Eames (1973)	CR<Am	
Powell and Dickson (1975)	Am<CR	1:2
Roberts <i>et al.</i> (1977)	Disperse Am <Spherical Am	
McCabe and Smith (1981)	Am<CR	1:2
De Gee <i>et al.</i> (1985)	Am<CR	
Lappalainen <i>et al.</i> (1989)	Am=CR	
Matsumura <i>et al.</i> (1995)	Two CR veneer materials (Dentacolor™)<(Visio-Gem™)	1:2

* Ratio compared with lower ranked material

CR: Composite resin

Am: Amalgam

In addition, there are some contradictions between clinical findings and laboratory results. For example, while the *in vitro* findings by O'Neal and Eames (1973) and Powell *et al.* (1975) showed greater rate of wear in amalgam than in composite resins, two clinical observations by Eames *et al.* (1974) and Phillips *et al.* (1973) both showed opposite results. Although it is difficult to predict clinical behavior of dental restorative materials on the bases of current *in*

vitro data, Sulong and Aziz (1990) stated that the best way to draw conclusions from these *in vitro* results may be the ranking of the tested materials when the study simulates the clinical situation as closely as possible. Recently, many attempts have been made to simulate oral environment using sophisticated wear machines (Ehrnford *et al.*, 1980; McCabe and Smith, 1981; Wilson *et al.*, 1981; DeLong and Douglas, 1983a; De Gee *et al.*, 1986; Roulet, 1987; Bailey *et al.*, 1988; Leinfelder *et al.*, 1989; De Gee and Pallav, 1994).

3.6.4 GIC wear studies

Few studies were made for the GIC wear studies. Several authors had studied clinical wear of conventional GICs but the results are not promising. Knight (1984) in his 27 month clinical study reported a 27% failure rate in Class II restorations with conventional GICs and that 35% of restorations showed severe wear affecting clinical function. Smales *et al.* (1990) reported only 57% of Ketac Silver restorations were rated as clinically acceptable, while two CRs and amalgam showed at least a 94% successful rate in their 3 year clinical study. Welbury *et al.* (1991) observed in deciduous teeth over 5 years, and failure rate was 33% while amalgam showed 20%. The median survival time was reported as 33.4 months and 41.4 months in Ketac-Fil and Amalgam, respectively.

Controlled studies of the clinical behavior of RM GICs used to restore occlusal cavity preparations in adult groups are relatively few (Smales *et al.*, 1990; Lidums *et al.*, 1993; Wilkie *et al.*, 1993). Obvious occlusal wear was not found at 12 months in immature permanent teeth in children which were restored with Fuji II LC (Maki *et al.*, 1994). However, the same authors reported wear rate after 12 months of up to 300 μm at the cavity margin when encapsulated Fuji II LC was placed in the occlusal preparations of permanent teeth in adults. Different resin-modified glass ionomers may vary in their resistance to occlusal wear. Croll (1994) found that Vitremer cements appeared to wear less than Fuji II LC and Photac-Fil over 2 years in Class I and II restorations.

3.6.5 Conclusion

The question arises as to whether the material showing minimum wear is a superior clinical dental material or not. In most situations, restorative materials begin to wear immediately after restorations are placed. In some cases, these restorations can accelerate tooth wear. In a clinical study by Monasky and Taylor (1971), they observed that natural teeth opposed by porcelain restorations showed excessive wear. Ideally, it is suggested that restorations should have similar wear characteristics to that of tooth structure to avoid loss of occlusal vertical dimension or over eruption of opposing teeth over time. (Heath and Wilson, 1976). The goal of

restorative dentistry should be provide occluding surface that not only resist wear but also do not wear opposing surfaces (Wiley, 1989).

Assessing wear is very difficult due to its multi-causative nature. For example, contacting load, contact duration, velocity of the movement, temperature and kind of lubricant are some of the important factors which influence the wear process *in vitro* (Czichos, 1981). In addition to these variables, from an *in vivo* perspective, there are other complicating factors such as the quality and quantity of saliva, food, consistency, and personal habits which all constantly vary (Dahl *et al.*, 1993).

Many attempts have been made to assess the wear characteristics of dental materials. Because *in vivo* experiments are often very complicated and expensive, many studies were made in an *in vitro* situation. However, there are no standard experimental procedures and devices and the results are contradictory. Although clinical results are the ultimate answer for the efficacy of the material, further research to develop *in vitro* wear experiments which simulate oral conditions are urgently needed.

3.7 Other assessments of dental materials

3.7.1 Testing of the bond strength

3.7.1.1 Adhesion to tooth structure

The major advance in GICs is its ability to absorb permanently to the hydrophilic surfaces of hard oral tissues, thus offering the possibility of sealing margins developed at the tissue interfaces during restorative procedures. Many hypotheses explaining the actual bonding mechanisms of GICs have been introduced (Smith 1968, Beech 1973, Wilson 1974, Wilson *et al.* 1983, Mount 1991). However, the precise nature of the adhesion of polyalkenoic cement to untreated dental enamel and dentine has yet to be established (Wilson and Nicholson, 1993). There is consensus that the mechanism for bonding to enamel is almost entirely a result of ionic and polar forces (Wilson and McLean, 1988). Wilson *et al.* (1983) studied the absorption of polyacrylate on hydroxyapatite using chemical methods. This confirmed that an exchange of ions occurred between the two components and they concluded that polyacrylate displaced surface phosphate and calcium, and entered the hydroxyapatite structure itself. This factor enabled them to postulate the existence of the intermediate layer of calcium and aluminum phosphates, and polyacrylates between the hydroxyapatite and cements. This intermediate layer was observed by Mount (1991), when the specimen was dehydrated. More recently, Lin *et al.* (1992) showed the ion-exchange layer between RM GICs and the dentine surface, using X-ray photoelectron spectroscopy and confocal microscopy.

3.7.1.2 Variation of the bond strength

There are numerous reports of the bond strength of dental materials. However, the published bond strengths indicate large inter- and intra- laboratory variations. One reason for these variations is the lack of a standardized test procedure. Even small modifications of the same method in the same laboratory can give 2 to 4 fold differences in bond strength values (Oilo and Olsson, 1990, Van Noort *et al.*, 1990). The FDI technical report (1990) concluded that the bond strength value is not as important as a comparison of values between materials, and the ranking of the bond strength values may be more meaningful than the actual values.

There are several factors which influence reported bond strengths. Firstly, there are differences between the two main test modes, namely, shear and tension. Secondly, there are a variety of designs of the test apparatus related to the alignment of specimens. Thirdly, the variables associated with the tooth substrate that have been cited to include different kinds of species, the time lapse from extraction, the storage medium, depth of cut of the dentine, and variations in surface preparation. Moreover, there is confusion in measurements of bond strength depending on whether it is cohesive failure or adhesive failure. In general, GICs are brittle materials, therefore, the failure often happens within the GICs, not at the interface of the tooth structure and GICs. This may explain relatively low bond strength of GICs, even when there is evidence of the ion exchange layer between tooth structure and GICs (Mount, 1991). Using the Scanning Electron Microscopy (SEM) analysis, it is easy to detect whether the fracture has occurred at the interface or at another level of the multicomponent adhesive system.

Recently, a standard adhesion test model was issued from the International Organization of Standardisation (ISO/ TR 11405, Dental Materials-Guidance on testing of adhesion to tooth structure, 1994). The purpose of this document is to 'standardise, as far as possible, different procedures whereby the effect or quality of a bond between a dental material and the tooth structure could be substantiated'. Although the standards may contribute to more comparable bond strength data between experiments, there is still no established correlation between these results and data from clinical trails. There are a number of factors which can result in different bond strengths *in vitro* and *in vivo*. There include, for example, contamination by bacteria, masticatory forces, dentinal fluid etc. Jemt *et al.* (1986) stated that the *in vitro* tensile bond strength of GICs to enamel is approximately twice as high as *in vivo* strength.

3.7.1.3 In vivo bond strength

Retention of restorations in cervical erosion and abrasion lesions has often been used to

evaluate whether a material successfully bonds to the tooth structure *in vivo*. Because of the non-retentive form of these erosion lesions and their frequency of occurrence, these kind of survival rate studies *in vivo* are relatively easy to observe. There is no invasive procedure involved for research, and it is a good way to access the durability of bonded restorations (Erickson and Glasspoole, 1994). However, there are several complications which influence their results, since the cavities may involve enamel, dentine or both, and in addition, the difference may depend on the age and habits of the patients. Table 3.3 and Table 3.4 summarize recent results of these retention rates of CR and GIC restorations. In spite of the fact that the bond strength of these GIC materials are reported as approximately 4-5 MPa while those of dentine bonded CRs are reported as approximately 15-20 MPa, the retention rates appeared to be similar for both restorations. Consequently, *in vitro* bond strength does not seem to predict the retention of these restorations. The reason for this may be that GIC materials with their low setting shrinkage, are not affected by sclerotic dentine as much as some dentine bonding systems.

3.7.1.4 Conclusion

As described before, there are many variables that influence bond strength, and the results often do not appear to correspond with the clinical results. In many studies, GICs are compared with CR materials and the bonding values of GICs are usually low compared to CR systems. The recent development of dentine bonding adhesives is promising, but currently, it is widely recognized that GICs are the materials that adhere chemically to tooth structure although the mechanisms involved are very complicated and not fully understood.

The favourable GIC retention rates *in vivo* suggest that currently available conventional bonding tests may not be an effective measure of the 'true' GIC bond strength to tooth structure. Moreover, many bonding tests may merely reflect the physical strength of the material with so-called 'adhesive failures' being 'cohesive failures' of the GIC due to its brittleness. Therefore, if the physical strength of GICs were improved, the apparent bond strength may increase. In this respect, careful investigation of the physical strength of GICs is of paramount importance.

Table 3.3
Summary of the percentage of the *in vivo* retention rate of composite resin restorations in cervical erosion /abrasion lesions

Authors	Bonding agent	Retention (%)			
		6months	1year	2years	3years
Duke <i>et al.</i> (1991)	Scotchbond 2	91	88	86	80
Bastos <i>et al.</i> (1991)	Scotchbond 2	93	91	85	70
Van Meerbeek <i>et al.</i> (1994)	Scotchbond 2	95	86	87	77
Taleghani and Leinfelder (1991)	Tenure	95	95		
Tyas (1992)	Tenure	87	87	70	72
Tyas and Chandler(1993)	Tenure	96	92	88	
Dickenson <i>et al.</i> (1994)	Universal		75		
Snuggs <i>et al.</i> (1994)	Allbond 2	100			
	Allbond 2		100		
	Epic TPTM		83		

Table 3.4
Summary of the percentage of the *in vivo* retention rate of glass ionomer restorations and 'sandwich' technique restorations in cervical erosion /abrasion lesions

Authors	Materials	Retention (%)				
		6months	1year	2years	3years	4years
Tyas and Beech (1985)	Fuji II	97	96	92		86
Tyas <i>et al.</i> (1989)	Sandwich technique			100		
Powell <i>et al.</i> (1992)	Ketac-Fil		97	97	97	
Powell <i>et al.</i> (1992)	Sandwich technique (dual cured GIC)			100		
Reich and Volkl (1994)	Ketac-Fil					96

3.7.2 Testing of the biocompatible factors

3.7.2.1 Introduction

Biological considerations are now very important when selecting and using dental materials. Knowledge of these factors has expanded significantly in the last two decades. Even if a material has superb mechanical properties, but is injurious to the pulp or soft tissue, it should be discarded. In a broad sense, the definition of a biomaterial is any substance that can be used for any period of time as a part of a system with no harm, and that the material should be stable for a long time in the oral cavity and the oral environment. Moreover, these materials should have no hazardous effects for the dentist, dental auxiliaries, or dental technicians (Phillips, 1991).

3.7.2.2 Microleakage studies

The hazard to the dental pulp from microleakage is often discussed. Studies of the pulp reaction to microleakage have advanced especially since adhesive materials, which were developed in the last two decades, have provided possible bonding to tooth structure. Microleakage tests have been used as one method of predicting the survival of adhesive materials *in vivo*. If adhesion to the tooth structure is not adequate, bacteria, food debris and saliva may influence the dentinal tubules or pulp by capillary action between the restoration and the tooth structure. This microleakage problem is considered as one of the possible causes of post-operative hypersensitivity. In addition, the microorganisms and the toxins they produce cause prolonged harmful effects to the pulp, and in the long term, can cause secondary caries or discoloration, and consequently, loosening or dislodgment of the restorations.

In recent years, the advent of new bonding systems related to CRs has led to a marked improvement in the marginal seal of restorations. However, especially in the early stage of development of these systems, the bonding did not provide a perfect seal between the tooth structure and the restoration. Failure occurs more often as time lapses and it is caused by the contraction from polymerization of dental materials, dimensional changes from hot and cold stimulation of both tooth structure and dental materials, dissolution of the materials and deterioration of the bonding strength after exposure to occlusal forces.

Testing microleakage *in vitro* involves either a contraction gap test or a qualitative/quantitative leakage test. The contraction gap test measures the maximum width of the contraction gap between the tooth-restoration interface a relatively short time after the materials have been cured. Often, epoxy resin replicas are used for this assessment and sometimes SEM imaging is

used. The leakage test uses dye penetration into the cavity after more than 24 hours have elapsed after placing the restoration. Qualitative microleakage tests are usually determined after thermocycling in the tracer, and the leakage is evaluated by the dye penetration at the margins of the restorations. Basic Fuchsin dye is commonly used, because of its ease of manipulation, and the scoring criteria for the microleakage is comparable to other studies (Sidhu, 1992). Eliades (1994) stated that silver nitrate is becoming increasingly popular as it utilises the advantages of the small atomic radius of silver, its low charge and low affinity to dental tissues compared with other ionic tracers. This technique provides a quantitative basis for measuring microleakage using SEM imaging. In addition to these common tests, there are several methods for evaluating *in vitro* microleakage. These include usage of radioisotopes, air pressure, bacterial penetration, pH changes and a neutron activation analysis method (Sidhu, 1992).

3.7.2.3 Composite resins versus GICs in microleakage studies

Numerous investigations of microleakage in composite resins have been reported, however, there are only a few studies comparing GICs with composite resin systems. Clinically, studies generally showed that GICs achieved better marginal seals than composite resin systems (Shimokobe, 1978; Mount, 1981; Fuks *et al.*, 1983; Aboush and Jenkins, 1986; Osborne and Berry, 1990; Tay and Lynch, 1990a,b). Tyas (1986) filled cervical abrasion regions with conventional GICs and composite resins, and concluded that GICs had favorable margins. Brandau *et al.* (1984) observed GIC fillings for 4 years and six months and reported that 9% of the cases showed discolored margins, but there was no discoloration which penetrated between the cement and the tooth structure and concluded that the marginal seal of GIC was satisfactory.

Alternatively, there have been a number of articles showing marginal leakage of GICs *in vitro* (Crim and Shay, 1987; Barakat *et al.*, 1988; Scherer *et al.*, 1989; Cheung, 1990; Mathis *et al.*, 1990; McInnes *et al.*, 1990). Because GICs are water-based cements, it is very important to select an appropriate dye, since apparent dye penetration with a water soluble dye may in fact be the result of dye diffusing into the bulk of the GIC, and not along the interface between the GIC and the tooth structure (Youngston *et al.*, 1990). Also, it is known that GICs are very sensitive to dehydration (Wilson and McLean 1988) and studies of gap formation need to consider the dehydration of the specimens to ensure that the results represent the true performance of these materials. Shono *et al.* (1993) demonstrated the usage of Cryo-SEM for observation of the microleakage of the resin hybrid layer. With this technique, specimens were placed in liquid nitrogen at -160°C and the interface was observed with SEM in a specially conditioned atmosphere. This method may contribute to more accurate GIC microleakage studies in the future. Soderholm (1991) suggested the usage of an appropriate dye which has a

molecular size similar to that of bacterial toxins. He also discussed the possible use of radiotraced bacteria to measure microleakage for GICs.

Recently developed RM GICs may be expected to react differently compared with conventional GICs, as polymerization shrinkage will occur in this new system due to the inclusion of the resin component. On the other hand, RM GICs have higher reported bonding strength than conventional GICs (Holton *et al.*, 1990; McCaghren *et al.*, 1990; Mitra, 1991). Most studies show favorable microleakage results *in vitro* with RM GIC (Torii *et al.* 1991; Hallett and Garcia-Godoy, 1993 Sidhu, 1994). In contrast to these studies, Douglas and Fundingsland (1992) reported no significant difference in marginal leakage when comparing dual-cured GIC liners and conventional GIC liners.

3.7.2.4 Correlation with in vivo microleakage

Although most *in vitro* microleakage studies seem to agree on the positive correlation between the microleakage and the bond strength of the restoration to the tooth structure, there is some controversy in the literature about this relationship. As time lapses, the bonded dentine surface can be partly affected by positive dentinal fluid flow. Such contamination could result in weak bonding in some areas, and microleakage occurs through these debonded areas even if another part of the surface still maintains a strong bond (Torstenson *et al.*, 1982; Soderholm, 1991).

Eliades (1994) suggested the need for a standardised microleakage testing method which sets the type and dimension of the cavity, marginal preparation, filling technique, finishing and polishing procedure, and type of tracer.

3.7.3 Erosion and leaching

Microleakage is not the only factor influencing the durability of restorations. Resistance to erosion and/or leaching may be important for the survival of restorations. However, the loss of soluble species from the set cement may be beneficial to the tooth structure. The release of fluoride by GICs is regarded as a clinically advantageous character although this reaction apparently takes place by ion exchange, therefore, no discernible loss of material is evident as the process occurs (Wilson and McLean, 1988).

Erosion results from both chemical attack and mechanical wear. In dentistry, the chemical attack comes from acids either present in foods and beverages, generated in the mouth by dental plaque, or from the stomach due to gastric reflux. To mimic this attack in laboratory testing, a static solubility test was originally carried out, which employed appropriate solutions

of erosive acids (Kent *et al.*, 1973). Recently, the mechanical aspect has been introduced (Wilson *et al.*, 1986b), using a test in which jets of aqueous acid impinge on a cement specimen.

A number of studies have been undertaken to assess the clinical relevance of this test. Beech and Bandyopadhyay (1983) concluded that the lactic acid jet test could be appropriate as it ranks materials in the same order of durability as that found clinically. According to this study, GICs were found to be more resistant to erosion than silicate with progressively more erosion in silicophosphate, zinc phosphate, and zinc polycarboxylate cements. This ranking has been confirmed by the other studies (Ibbetson *et al.*, 1985; Wilson *et al.*, 1986a).

More recently, the ISO adapted this impinging jet acid erosion test using aqueous lactic acid as one of the standardizing tests for water-based cements. Billington *et al.* (1992) examined 20 commercially available glass ionomer cements from 6 manufacturers using this method. The results showed a wide range of erosion rates, with generally lower rates for luting cements than restorative materials, though little could be deduced about the factors which influence of the erosion rate. In general, this test is rarely discussed in the literature, and currently, the effect of longer maturation time of both conventional and RM GICs upon erosion resistance is unknown (Williams *et al.*, 1992). Thus, further research is needed on these aspects.

3.7.4 Toxicity of GICs

Some controversy exists regarding pulp reactions and post-operative problems with the use of GICs (Klausner *et al.*, 1989; Johnson *et al.*, 1993). Some researchers concluded that the pulpal response was correlated to the bacterial leakage (Plant *et al.*, 1988). However, the study by Paterson and Watts (1987) suggested that pulpal response still existed on germ-free rats. While mechanical pressure (Pameijier and Stanley, 1984) was suggested as one of the causes of the post-operative sensitivity, Heys *et al.* (1987) could not confirm this observation when cementing crowns on monkey teeth.

The inclusion of the resin component resulted in the existence of monomers in GICs, such as HEMA in the freshly mixed material (Wilson and Nicholson, 1993). This may cause new toxic reactions which were not found in conventional GICs. Janeckova *et al.* (1989) reported that patients who had an allergic reaction to non-metal materials (e.g. epoxy resin, rubber) also had a reaction with HEMA particles. They reported that the patients reacted because of a reduced ability of neutrophils to exert phagocytic action on HEMA particles and a decrease in levels of alpha-2-macroglobulins in serum. There is little information available about the toxicity to the RM GICs and more research is necessary.

3.7.5 Conclusion

The usage of RM GIC should be considered and examined carefully, because it may effect the development of the ionic exchange layer between the restoration and tooth structure due to the inclusion of resin, and the subsequent marginal seal against the tooth structure needs further evaluation. Biocompatibility of RM GIC may be compromised as its resin component may be harmful to the oral tissues. These materials are reported superior in some properties, such as mechanical properties and bond strength, however, an assessment of the long term durability of dental products as biomaterials needs to be undertaken carefully in the oral environment.

3.8 Conclusion

In the ideal situation, whenever a new dental product is introduced into the market, 'all the possible investigations' should be conducted thoroughly on the new dental material. 'All the possible investigations' means not only thorough in *in vitro* assessment, but also 'all the possible investigations' of every clinical situation. Although it is obvious that the initial evaluation of a new product should include *in vivo* assessment, this is often more difficult than *in vitro* study as it is more time consuming and expensive, and of dubious ethical standing. Also, it is sometimes difficult to obtain meaningful results *in vivo* as clinical situations vary from patient to patient, tooth to tooth, condition to condition, and operator to operator. That is, there are no standard conditions in clinical situations, therefore, no data applies in the same way in each clinical situation and many variables influence the success of procedures. Therefore, although it is of paramount importance to obtain clinical results, *in vitro* data are more abundant and often used as the basis for primary evaluation by clinicians. This tendency is more evident where the speed of development of new materials is accelerated. Often clinical data does not appear in the literature until after a number of dentists have been clinically using a material for quite a while. Naturally, while there is no doubt that clinicians make efforts to get as much information as possible before they start to use the new materials, it is a fact that they often have to rely on their judgment of the *in vitro* information that manufacturers provide especially in the early stage of a material's usage.

The question also arises as to whether this early data from manufacturers is truly reliable or not. Many of the manufacturers' data are collected using tests following the ISO standards. While the existence of standard tests makes comparison among materials and among studies easier, it should be realized that having ISO standards does not guarantee satisfactory clinical results because the standards often merely provide the basic methodology for testing. Also, given the recent large range of dental materials with complex properties, choosing appropriate tests poses

some difficulties. The best standard test to judge one material may not be very applicable to judge other materials. Consequently, clinicians should be careful in evaluating new materials based on these data, and it is important to identify useful experimental methods which are more clinically relevant rather than rely on the routine standard test methods which may lack clinical significance. Ultimately, the final decision on the use of any product or technique rests with the dentist (Simonsen, 1995b).

Despite the limitations, *in vitro* tests can give significant information which predict the clinical behaviour of dental materials. Ideally, the conditions under which *in vitro* testing is carried out should simulate as closely as possible to the clinical situation for the results to have clinical relevance. Currently, there is no *in vitro* test which simulates the clinical situation, however, attempts to reproduce the oral environment should be further developed, and more research will be needed to understand the anatomical and biochemical aspects of the dentine and enamel themselves to correctly comprehend these results.

Part I -Strength of GICs compared to CRs

Chapter 4 Preliminary Studies on Shear Punch Tests

4.1 Overview

The aims of this part of the study were to assess the validity of the shear punch tests and to establish a protocol for the shear punch tests. Because this study involved the development of a non-standardised physical strength test, a series of preliminary studies were conducted to control and minimize errors accompanied with the specimen preparations and the experimental conditions for both CRs and GICs. The following preliminary tests were conducted:

Preliminary Study One: Reproducibility of the shear punch test results

Preliminary Study Two: Coating of GICs and the shear strength

Preliminary Study Three: Storage condition and the shear strength

Preliminary Study Four: Light cure time and the shear strength

Preliminary Study Five: Maturation time of GICs and CRs

Based on this information, protocols of the final experiments were formulated. They will be discussed in detail in Chapter 6.

4.2 Introduction

The importance of testing physical strengths of dental restorative materials is obvious. Immediately after materials are placed in the oral cavity, they are exposed to relatively high stress due to the mastication. On some occlusal surfaces, for example, up to 193 MPa compressive stress could be applied (Phillips, 1991). Strength is the maximal stress required to fracture a structure. Numerous reports of mechanical strengths of restorative materials by various researchers in various conditions have been published. However, the shear punch tests, that are extensively discussed in this study, are less well-known than the traditional physical strength tests. There are standardized ISO tests, but different tests are specified for different materials such as GICs and CRs and that makes it difficult to compare the materials (for CRs; flexural test, ISO4049:1988; for GICs; the compression test, ISO 9917:1991). In addition,

there are some concerns about the required size of specimens for the standard physical strength tests. Mount *et al.* (1994) stated that the shear punch test has several advantages which are evident during construction of the specimens. They were described as:

- built from a single mix and not being layered.
- thin enough to allow total cure through the full depth of the specimen by light activation.
- small enough to be fully activated with single application of the light activator (no multiple irradiation).
- simple construction and economical to construct.

The standard flexural test for composite resins requires the size of (25 ± 0.2) mm x (2.0 ± 0.1) mm x (2.0 ± 0.1) mm (ISO 4049,1988). The specimen mould of the standard compression test for GICs is the size of 4.0 mm in diameter and 6.0 mm in height (ISO 9917,1992). Both specimens are relatively large so that the preparation of the light-cured materials needs multiple irradiation due to the limitation of cure. This can result in areas with different degrees of cure in the same specimen. Mount *et al.* (1996) stated that the shear punch test appeared to be more satisfactory than the standard tests set by ISO for the estimation of 'real' physical strengths of these aesthetic restorative materials, although there is little published in the literature to support this hypothesis (Mount *et al.*, 1994, Mount *et al.*, 1996).

In this chapter, the shear strengths of a range of GICs and CRs were tested under different conditions. Based on the findings of this chapter, the final protocol for the shear punch test described in the rest of the chapters was established.

4.3 Materials and methods

4.3.1 Materials

Five GICs and two composite resin restorative materials were investigated in the experiments described in this chapter. The individual materials used, along with the respective manufacturers, batch numbers and codes used are shown in Table 4.1.

Of the five GIC groups, Fuji II LC Blue, Fuji IX and Vitremer were hand-mixed versions. HiDense and Miracle Mix were capsulated versions. HiDense was described as "glass ionomer silver reinforced restorative (Capsules)" by the manufacturer. Miracle Mix was described as an 'auto-cured GIC with the inclusion of the metal component in the powder' according to the manufacturer's information. Both Fuji II LC Blue and Vitremer were RM GICs. The tested CRs were Z 100 and Pertac Hybrid and both were light-cured materials. They were described as hybrid composite resins by the manufacturers. In Preliminary Study Two (Coating effect of

GICs and the shear strength), one varnish and one unfilled resin were used for coating purpose. The materials were all widely available in Australia except for Fuji II LC Blue which was a trial material from GC (Tokyo Japan).

Table 4.1
Materials and manufacturer's details for the shear punch tests in this chapter

Products	Manufacturer	Batch #	Description/ mixing type
<u>GICs</u>			
Fuji IX	GC Co., Tokyo Japan	110341	C GIC/powder-liquid
HiDense	Shofu, Kyoto Japan	931027-1	CM GIC/capsulated
Miracle Mix	Shofu, Kyoto Japan	920114	CM GIC/capsulated
Fuji II LC Blue	GC Co., Tokyo Japan	050441	RM GIC/powder-liquid
Vitremer	3M Dental, St. Paul, USA	19930223	RM GIC/powder-liquid (A3 shade)
<u>Composite resins</u>			
Z100	3M Dental, St. Paul, USA	19940222	H CR (A3 shade)
Pertac Hybrid	ESPE, Seefeld, Germany	0067X301	H CR (A3 shade)
<u>Coating</u>			
Fuji Varnish	GC Co., Tokyo Japan	301171	
Ketac-glaze	ESPE, Seefeld, Germany	0076Z028	

C GIC: Conventional GIC

RM GIC: Resin-modified GIC

CM GIC: GIC with inclusion of the metal component

H CR: Hybrid composite resin

4.3.2 Methods

A modified technique based on the methods described by Mount *et al.* (1996) was used in this study. For most experiments, the methods involved the protocols described in this chapter. In experiments where special procedures were followed, they have been described in the specific 'materials and methods' section.

Specimen preparation

Brass washers approximately 18 mm in diameter 0.8 mm thick were used for the specimen moulds. Each washer had a internal 6 mm diameter hole into which the materials were placed. A jig to align these washers was made with 1.0 mm thick microscope slides, and a mylar strip was placed on the lower surface to separate the materials and the slide (Fig 4.1). The capsulated materials were prepared according to each manufacturer's instructions. In powder/liquid type GICs, the materials were dispensed according to the manufacturer's suggested powder/liquid ratio (Fuji IX: 3.6/1; Fuji II LC Blue: 3/1; Vitremer: 2.5/1) using an electronic balance (AC-400, Phoenix, Denver, USA). The standard capsule for mixing GICs held sufficient material for two specimens. For the powder/liquid type GICs, three GIC specimens were generally made from one mix of the materials. The materials were placed in the mould with a Centrix syringe (Centrix Inc, Shelton, USA) and covered with a second mylar strip. A glass slab was placed on the mylar strip to level the materials. Ten specimens of each material were prepared at a single time as one series.

For light-cured composite resin and resin-modified GIC materials, the jig was inverted and the glass slide was removed. The diameter of the specimens was less than the diameter of the exit window of the irradiation unit so that the materials could achieve the maximal activation with a single application of the curing light unit. Using the light curing unit (Translux CL, Kulzer gmbH, Friedrichsdorf, Germany) with a 7.0 mm diameter light outlet, the specimens were cured from the reverse side for 40 seconds through a mylar strip. For conventional GICs, the mylar strips were left on both sides of the specimens to maintain the water balance and removed after they reached their initial set (approximately 15 minutes). They were then immediately stored in water unless particularly specified. Specimens were stored in air tight containers for the time specified by the experimental conditions.

After storage in water, excess materials were roughly trimmed using a knife, and the brass washers were ground for approximately 45 seconds against wet 320 grit carborundum paper in a small polishing machine (Minimet, Buehler Co. Illinois, USA) to reduce the thickness to approximately 0.8 mm (Fig 4.2). After this treatment, both surfaces of the specimens were flat and the two surfaces appeared parallel. The thickness of the specimens was measured with a micrometer (Moore and Wright, Sheffield, England), and recorded before punching (Fig 4.3).

Setting up the shear punch device

The Hounsfield Tensometer (Fig 4.4) and the attached recording drum with a heat sensitive paper were prepared. The test apparatus consisted of a 3.2 mm diameter punch and corresponding die (Fig 4.5). The specimens were placed onto the tensometer with the support of Bluetac™ and the punch was advanced through it in a compression cage with a crosshead

speed of 2.0 mm per minute. The load at fracture of each specimen was measured from the record on a heat sensitive paper. The specimens which showed clear surface defects before punching or those which showed early fractures while punching were discarded from the group. The shear strength was calculated from the equation:

$$\text{Shear strength (MPa)} = 9.807A / \pi DB$$

Where A was load at fracture (kg), D was the diameter of the punch (mm), B was the thickness of the specimen (mm) after polished. However, D was fixed as 3.2 mm in this study, thus, the formula was simplified as below:

$$\text{Shear strength (MPa)} = 0.9760 A / B$$

Based on this formula, the strengths of each material were calculated. The mean strengths and the standard deviations were calculated using the Apple Macintosh version of STATVIEW 512+™ Version 1.0 (Abacus Concept Inc).

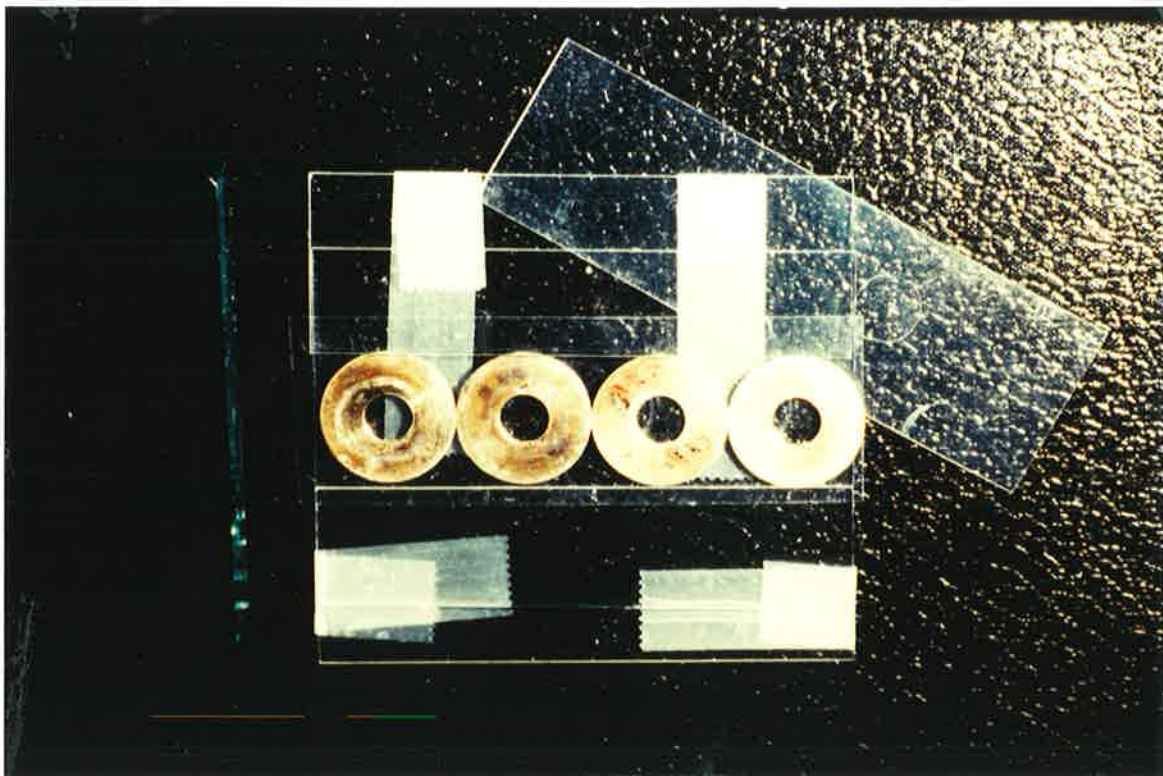


Fig 4.1

A view of an aligned jig. Four brass washers for the shear punch test. A mylar strip was placed on the lower surface to separate a slide and materials.



Fig 4.2

A view of a specimen in Minimet machine. A specimen was ground against wet 320 grit carborundum paper to reduce the thickness to approximately 0.8 mm.



Fig 4.3

The thickness of the specimen was measured with a micrometer and recorded before punching.

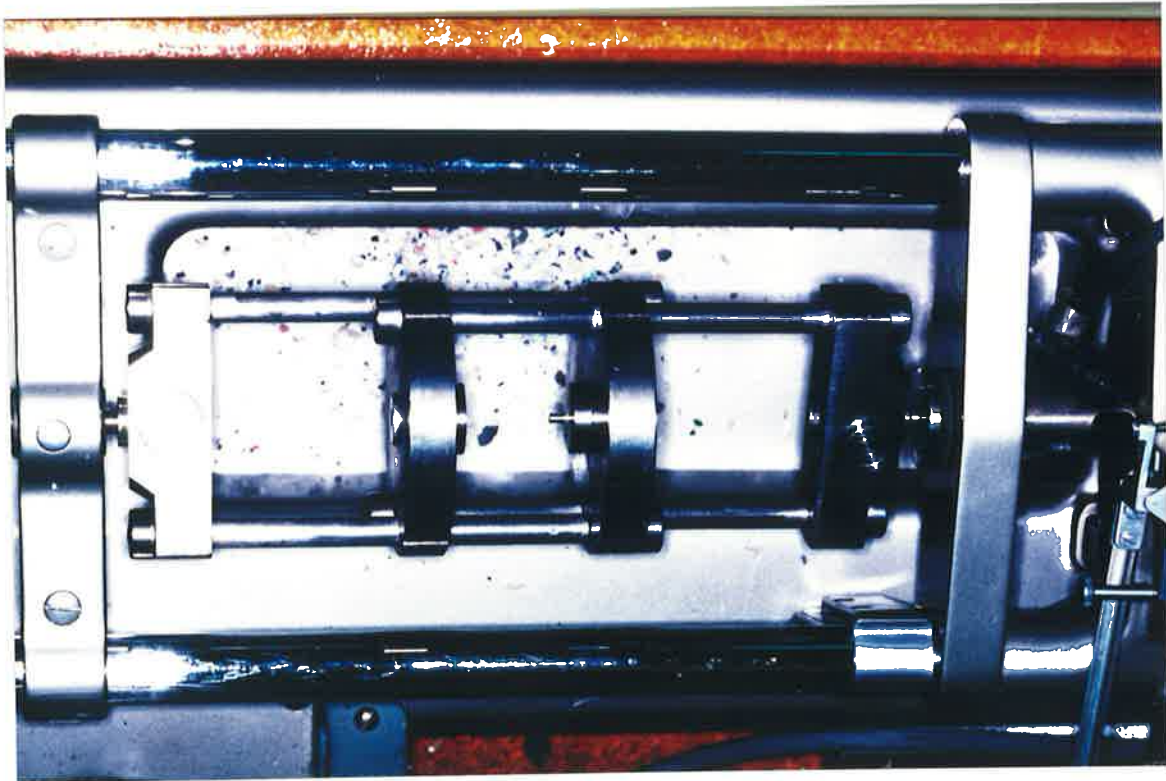


Fig 4.4

The lateral view of Hounsfield Tensometer showing a punch set up in the compression cage. The punch was advanced through with a cross-head speed 2.0 mm per minute.

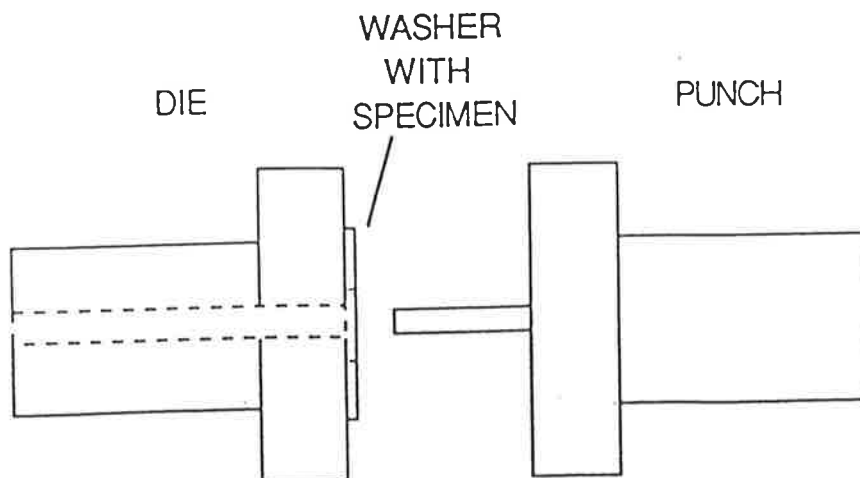


Figure 4.5

Diagram of a setting of a specimen and a die and punch unit. A punch is an exact fit in the hole of the die.

4.4 Preliminary Study One- Reproducibility of the shear punch test results

4.4.1 Introduction

The shear punch test was originally developed by Roydhouse (1969) as previously described (see Section 3.5.3.2). Although the shear punch test is not the standard test for GICs and CRs, it appeared to be an appropriate alternative for compression and flexural strength tests (Mount *et al.*, 1994). The shear punch test has the advantage when used in evaluating light-cured/dual-cured materials of requiring relatively compact specimens (approximately 1.0 mm thick and 6.0 mm in diameter) allowing that light penetration to be achieved. Mount *et al.* (1996) examined the shear strength of several GICs and CRs in their study and concluded that ten specimens are probably sufficient to achieve acceptable results in any series because of the low standard deviation and high reproducibility. The first preliminary experiment was made to determine whether a sample size of 10 was sufficient to demonstrate differences between materials.

4.4.2 Materials and methods

According to the procedures described in section 4.3.2, ten each of Fuji IX and HiDense specimens were prepared. They were stored in water for one week and the strengths were tested in each material (Group I). The same procedures were repeated after approximately three months (Group II) and the results of a total of forty specimens were compared using a two way ANOVA test.

4.4.3 Results

The results of the shear strengths in this part of the study are shown in Fig 4.6 and Table 4.2. Table 4.3 shows the result of the statistical analysis. These results suggested that there were significant differences between the materials ($p < 0.05$), but that there were no significant differences between Group I and Group II ($p > 0.05$).

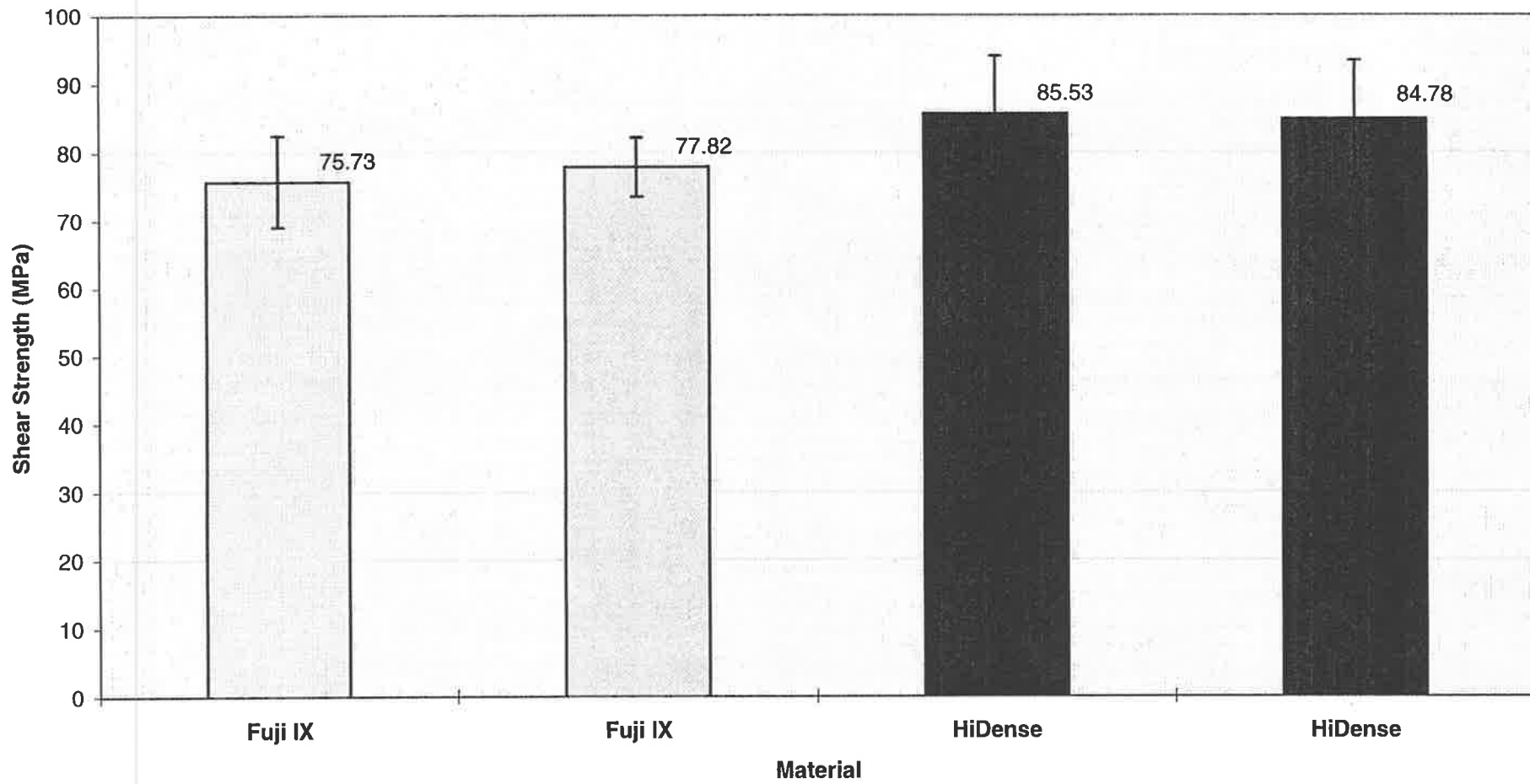


Figure 4.6
Preliminary Study One Reproducibility of the Shear Punch Test Results
The mean shear strength of Fuji IX and HiDense (two groups of each) stored in water for one week.
* Error bars represent the standard deviation.

Table 4.2

The results of Preliminary Study One. The sample sizes, the mean strengths and the standard deviations of each group of Fuji IX and HiDense specimens.

Group*	Materials	N**	Mean strength (MPa)	SD
I	Fuji IX	10	75.73	6.72
II	Fuji IX	10	77.82	4.34
I	HiDense	10	85.53	8.44
II	HiDense	10	84.78	8.54

Group*: The specimens of Group II were prepared and tested in the same manner as those of Group I after 3 months later.

N*: Number of the specimens

Table 4.3

The results of the statistical analysis of Preliminary Study One. (Two way ANOVA test)

Source	df*	SS**	F ratio	P value
Groups (A)	1	4.48	0.07	0.079
Materials (B)	1	701.83	11.05	0.002
AB interaction	1	20.24	0.32	0.576
Error	36	2286.19		

df*: Degree of freedom

SS**: Sum of Square

4.4.4 Discussion

The results showed relatively small standard deviations (5-10% of the mean values). The strengths of the specimens made of the different material showed significant differences ($p < 0.05$) while the strengths of the specimens between Group I and Group II did not show significant differences ($p > 0.05$). This suggested that the shear punch test results were reproducible and tests with a sample size of ten were adequate to demonstrate differences in the strengths of the two materials. This finding supported the statement by Mount *et al.* (1996) who examined the strengths of some GICs and CRs using the shear punch test. They found that the result obtained from the thirty specimens was very similar to that from randomly selected ten specimens out of the thirty specimens. They suggested that a sample size of ten was adequate to represent each materials' strength using this method.

The high reproducibility of the shear punch test results could be explained in the following way. Firstly, the simple procedures to construct the specimens. The small specimen size meant they could be constructed in a short time which was beneficial in the case of GICs because chance of dehydration while preparing the specimens was less compared to larger sized specimens required in other tests. This size also minimized the possible variations due to the incremental technique required to construct a larger specimen. For example, it was possible to make four specimens from one mixing procedure for Fuji IX, and two from one HiDense capsule.

Secondly, the simplicity of assessing data. As described previously, the formula to derive the shear strength in this study was:

$$\text{Shear strength (MPa)} = 0.9760 A / B,$$

where A is load to fracture (kg) and B is thickness of the specimen (mm).

Both values, A and B were easy to measure and were not operator sensitive. Also, this test allowed for the variation in the thickness of the specimens, while most physical strength tests require specimens with a particular size.

Thirdly, the loading area was very small and the materials were evenly supported by die metal without concentration of the stress in particular areas such as seen in some tensile strength tests (see Section 3.5.2). In addition, the influence of the surface flaws was small due to the fact that the materials were well supported. Mount *et al.* (1996) stated that there was less influence of the defects such as small pores and voids in the shear punch tests than in flexural bend tests and diametral compression tests. In most of the physical strength tests, crack formation can be

initiated by such surface flaws when the specimen is loaded and this kind of early fracture could be recorded as inconsistent data with lower physical strength.

4.4.5 Conclusion

The shear punch tests appeared to provide consistent, reproducible data which might be used as the one indicator of the physical strength. Ten specimens of each materials produced sufficient information to identify each material's physical character.

4.5 Preliminary Study Two-Coating of GICs and the shear strength

4.5.1 Introduction

It has been generally stated that early moisture contamination of conventional GICs causes lower physical properties (Wilson *et al.*, 1979, Mount and Makinson, 1982). This is related to the complex setting reaction and the effect of water. During initial gelation, water weakens the bonds between molecules, reducing the final strength of the hardened cements (Saito, 1993). However, without sufficient moisture, the continuous metallic-salt formation is inhibited, and the cement dehydrates and crack formation starts.

Thus, to maintain the moisture balance, isolation with varnish or low viscosity unfilled resin was recommended during the water sensitive stage of conventional GICs. Earl *et al.* (1985, 1989) examined the effect of different coatings using radioisotopes and found that low-viscosity light activated resin bonding agent showed the best sealing result. Mount (1994b) stated that the varnish provided by the manufacturer was not a good material to seal conventional GICs as it had an evaporative vehicle and porosity was likely to appear once the vehicle evaporated. Other researchers reported that low viscosity nail varnish provided the best sealing *in vitro* (Serra *et al.*, 1994).

The purpose of this preliminary study was to compare the shear punch results of the conventional GIC specimens in a coated and non-coated condition.

4.5.2. Materials and methods

A total of thirty Fuji IX specimens were prepared. For the first ten specimens, both sides of the materials were covered with Ketac-glaze and each side was light cured for 20 seconds immediately after mylar strips were removed after initial set. For the second ten specimens, after initial set, mylar strips were removed and both sides were sealed with Fuji Varnish then lightly dried. A second Fuji Varnish coating was applied to the both sides. After coating, all specimens were stored in water for one week. The last ten specimens were the control group which were stored in water without any coverage after initial set. The details of Fuji IX and the

coating materials used in this section are listed in Table 4.1. All the specimens were ground prior to the test in the same manner described in Section 4.3.2.

4.5.3 Results

The results of the shear punch test for each group are shown in Figure 4.7 and Table 4.4. The results of the statistical analysis are shown in Table 4.5. There were no significant differences in shear punch strengths between the groups ($p>0.05$).

Table 4.4

The results of the shear punch tests of Preliminary Study Two. The sample size, the mean strengths and the standard deviations of the Fuji IX specimens with different coating conditions.

Condition	N*	Mean strength (MPa)	SD
Ketac-glaze	10	71.12	6.96
Fuji Varnish	9	70.16	3.00
Control (no coating)	9	75.64	4.34

N*: Number of the specimens

Table 4.5

The results of the statistical analysis of Preliminary Study Two. (One way ANOVA test)

Source	df*	SS**	F ratio	
Between groups	2	115.58	77.79	p=0.071
Within groups	25	658.68	26.35	
Total	27	814.26		

df*: Degree of freedom

SS**: Sum of Square

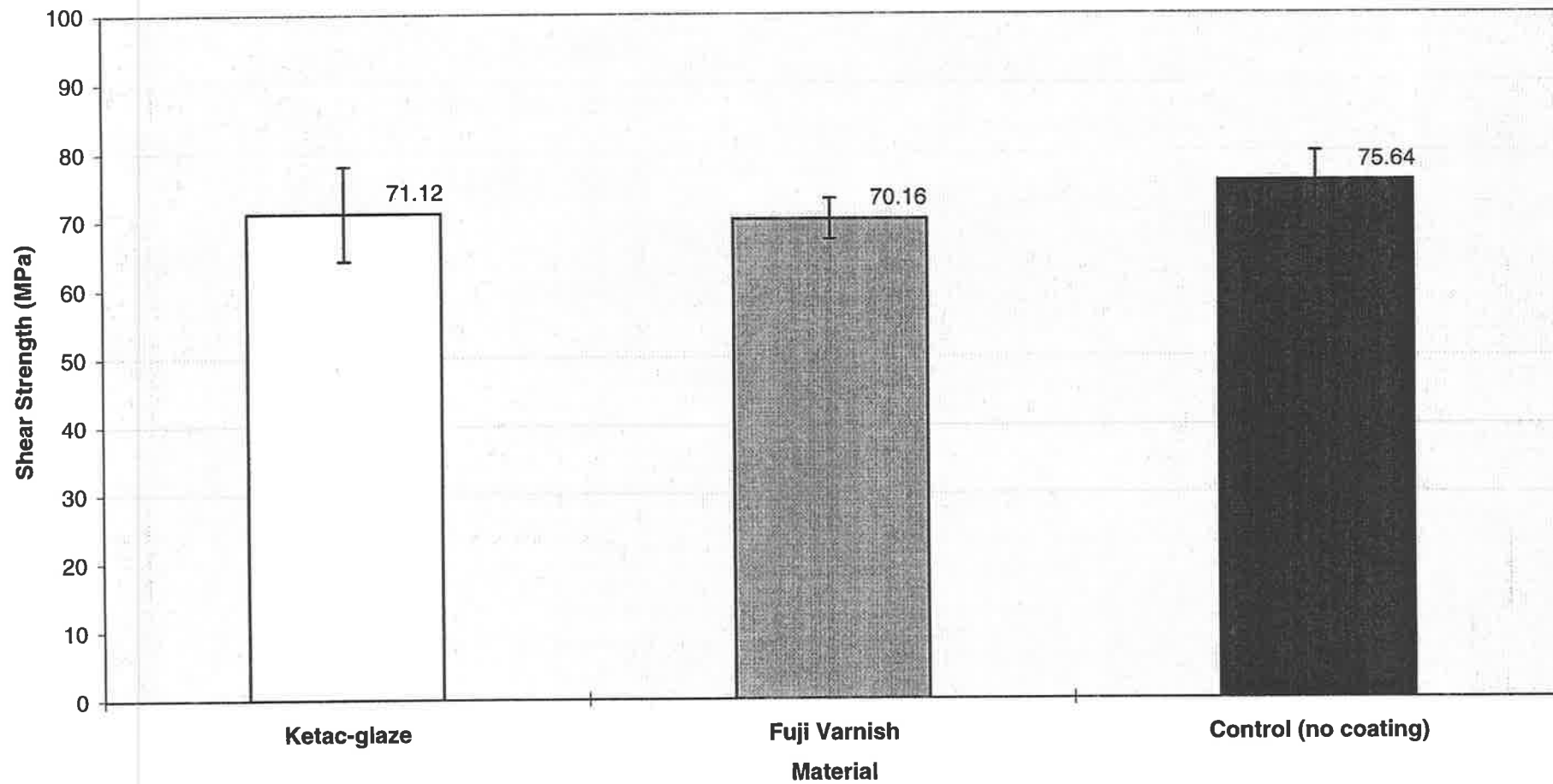


Figure 4.7
Preliminary Study Two Coating Effecting of GICs and Shear Strength
The mean shear strength of three groups of Fuji IX stored in water for one week.
* Error bars represent the standard deviation.

4.5.4 Discussion

The complete setting reaction of GICs is not clear, but it is explained in the following way (Wilson *et al.*, 1981). GICs set and harden by a transfer of leachable metal ions from the glass to the polyacrylic acid, to form a salt hydrogel. This initial setting reaction (gelation) is regarded as the result of chain entanglement as well as weak ionic cross linking (Wilson and McLean, 1988). Water is a by-product of the setting reaction and is an essential part of the hydrogel. The initial matrix-forming metal ions (calcium polyacrylate chains) are in a soluble form and vulnerable to attack by aqueous fluids. However, the sensitivity to water decreases as the cement matures due to progressive cross linking which replace hydrolytically unstable calcium polyacrylate with more stable aluminum polyacrylate. In conventional GICs, several researchers have supported this theory and reported that once a certain stage has been passed, the material becomes more tolerant to hydration and dehydration (Saito, 1993, Mount, 1994b).

Clinically, the need to coat GICs in their early stage of the setting is well established (McLean, 1988; Earl *et al.*, 1989; Haddad *et al.*, 1992). Too early exposure to water results in loss of the substance, causing a "chalky" surface and discoloration (Burgess *et al.*, 1995). However, several questions arise about this procedure. Firstly, how much premature exposure to extrinsic water influences the properties and how much it influences to the curing reaction of cements is not well defined in the literature (Shen and Grimaudo, 1994). Secondly, it is not clear whether the water contamination influences only the surface of the GIC restoration or whether water can penetrate and result in deterioration of the whole restoration. Thirdly, it is also not clear how long GICs should be protected from the moisture. Several researchers have suggested various critical times. This ranges from the first 15 minutes to the first 24 hours to show clinical maturity sufficient to be polished (Mount, 1994b). Shen and Grimaudo (1994) suggested 30 days isolation as enough time to minimize the effect of the water according to their *in vitro* study, but this is not always realistic in the oral environment where it is hard to keep a restoration from exposure to food, saliva etc. for prolonged periods of time.

In the clinical situation where GIC luting cements are used, it is in fact not always easy to apply coating to the every part of the restoration margins. It is particularly difficult when the margins of the restoration extend into a proximal surface or a subgingival area. Even if GICs are successfully coated, it can create a possible problem with the residue of these coatings in the subgingival sulcus where it may be difficult to remove. Mount (1994b) stated that the first setting cements such as Type I luting cements were less susceptible to water after initial setting so that there was no need for coating. To the contrary, some researchers have suggested that coating is important to prevent marginal dissolution while the setting reaction continues

(Kawakami and Shimokobe, 1993; Fruits *et al.*, 1996).

It has been argued by several researchers that absorption of water by GICs is clinically beneficial. It was found that GICs underwent volumetric contraction during the setting process (Feilzer *et al.*, 1988; Grajower and Guelman, 1989). Hinoura *et al.* (1993) stated that water sorption and subsequent swelling of GICs due to their hydrophilic character may lead to partial compensation of this shrinkage. Feilzer *et al.* (1995) agreed with this statement and examined the setting stress of both conventional and resin-modified GICs, and stated that early exposure to water led to stress relief and prevented spontaneous fracturing. However, these kinds of properties may not be clinically beneficial as the effect of exposure of the material to moisture may improve one property of the material such as contraction shrinkage, but can result in deterioration in other properties such as the strengths or esthetics.

It was expected that coated specimens would show higher strengths than non-coated specimens because of the early moisture contamination. However, in this preliminary experiment, it was interesting to observe that although there was no significant difference ($p > 0.05$), the shear strengths of non-coated specimen showed slightly higher values than those of coated specimens. There are no clear explanations for this result, but it may be that the cements set relatively fast so that the influence of the water was minimal. Another explanation may be that the possible residues of the coating materials on the surfaces of the coated specimens influenced the strength because the strengths were calculated according to the thickness of the specimens.

In visual observation, the one week old uncoated specimens did not show any surface deterioration, color change or other differences compared to the uncoated specimens. All these data appeared to support the fact that non-coated Fuji IX specimens did not have significant deterioration in this experimental condition where the specimens were exposed to water approximately 15 minutes after mixing.

However, the results of this preliminary study should be interpreted within the context of the specific experimental conditions. The same material could behave differently in the oral environment because the influence of saliva may be different from that of distilled water. Also, different GICs can behave differently. The influence of water varies depending on the chemical makeup of the materials. Especially RM GICs were found to resist water dissolution better after light-activation than conventional GICs presumably due to the early setting resulting from the inclusion of the resin component (Um and Oilo, 1992). In addition, some modifications such as

the use of the glass particles from which surface excess calcium ions were removed (Schmidt *et al.*, 1981) may result in minimizing the water sensitivity at the early setting stage.

More molecular level research will be necessary to understand this matter, and further research investigating the setting reaction of GICs would be certainly helpful to explain the relationship between early water contamination and the chemistry of GICs.

4.5.5 Conclusion

In this study, there was no significant difference found in the strengths among the coated GIC specimens and uncoated GIC specimens.

4.6 Preliminary Study Three-Storage conditions and the shear strength

4.6.1 Introduction

The influence of water contamination at the early setting phase of GICs was discussed in the previous preliminary experiment. Although the clinical benefits related to the coating are generally well accepted, a question arises about the influence on the *in vitro* mechanical property testing due to the extra-thickness of the coating materials. All strength testing is thickness-sensitive, and coating several times may contribute to the extra-thickness and consequently may influence the strength. In addition, 'laminating' the materials with a thin layer substance may change performance of the materials.

Some researchers have attempted to overcome this problem by storing GIC specimens in 100% relative humidity prior to the mechanical strength tests (Mittra and Kedrowski, 1994; Suzuki *et al.*, 1995; Mount *et al.*, 1996). In this condition, direct contact with water is avoided, and therefore it could be argued that applying the coating materials to the testing GIC specimens was unnecessary. The moisture in a humidior was assumed to maintain the moisture balance in the cements in this case.

The purpose of this preliminary experiment was to investigate whether this hypothesis was acceptable or not under the condition of the present study.

4.6.2 Materials and methods

Twelve groups of ten specimens each were prepared in the manner described in Section 4.3.2. After initial set, ten specimens each of Miracle Mix, Fuji IX, Vitremer and Z100 were stored in an air-tight humidior with 100% relative humidity at room temperature (Condition I). Another ten specimens of each material were stored in water at room temperature (Condition

II). They were stored under these conditions for one week before the shear punch tests were carried out.

This procedure was repeated on twenty specimens of both Miracle Mix and HiDense. In each case ten specimens were stored both in Condition I and II, but the ten of Miracle Mix specimens were tested after 2 hour storage time, and HiDense specimens were tested after 6 week storage time.

4.6.3 Results

The results of the shear strength of this experiment are shown in Figure 4.8, Table 4.6 and Table 4.7. Differences between the samples were assessed using a two way ANOVA test.

Most of the specimens in Group I (in humidior) showed lower physical strengths than those of Group II (in water) while one week old Miracle Mix and Vitremer specimens showed the similar strengths. There were significant differences between both the materials and the conditions ($p < 0.05$).

On visual examination, the edges of some specimens in Group I showed a whitish colour change similar to that which is often seen in dehydrated GIC restorations. Based on this information, the specimens of 100% relative humidity at room temperature appeared to show evidence of desiccation. None of the specimens showed clear crack formation.

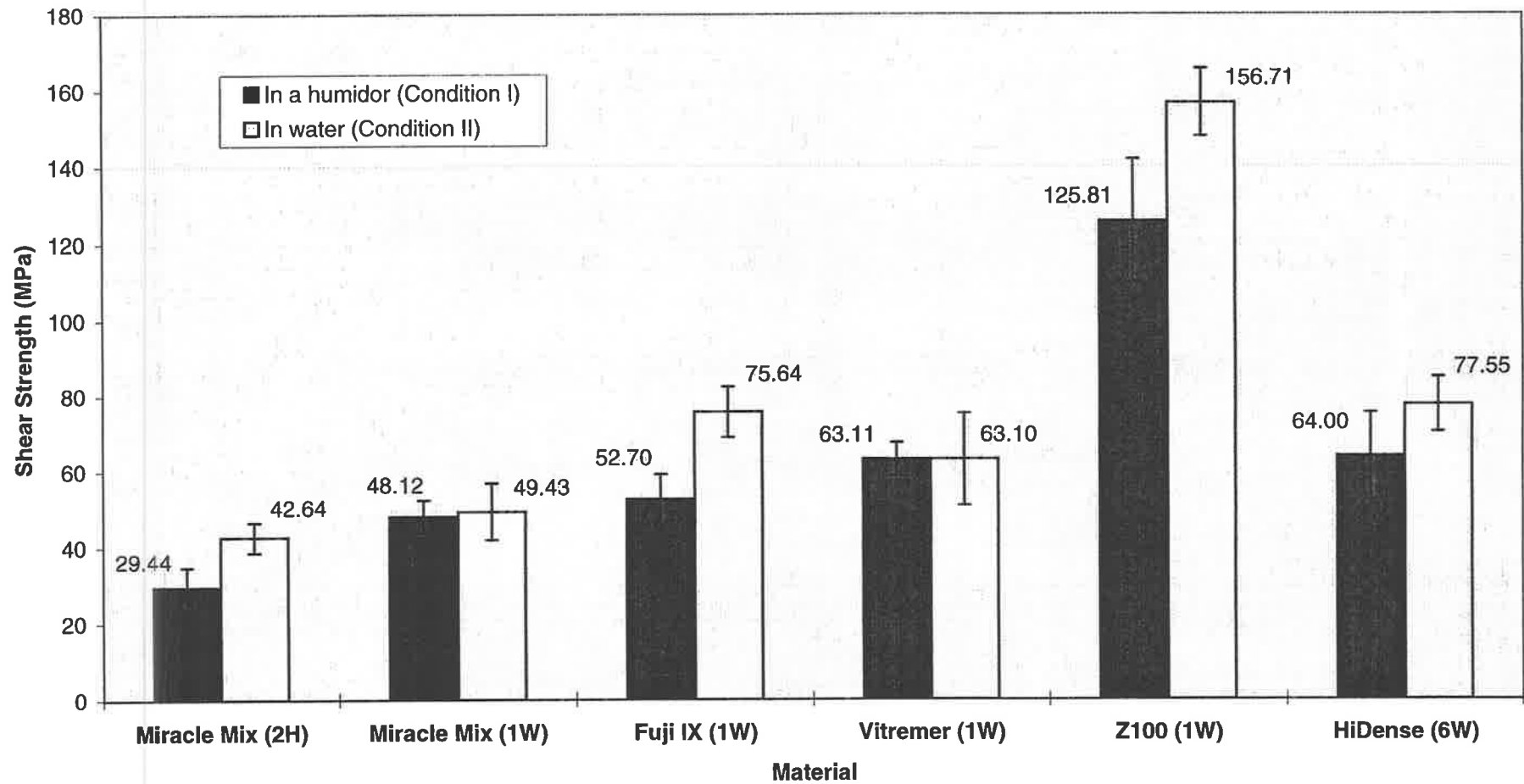


Figure 4.8

Preliminary Study Three Storage Conditions and Shear Strength

The mean shear strength of five GICs and one CR stored in water and 100% relative humidity at room temperature.

* Error bars represent the standard deviation.

Table 4.6

The results of the shear punch test of Preliminary Study Three. The mean strengths and standard deviations of the each specimens.

Condition*	Materials	N**	Mean strength (MPa)	SD
I (2h)	MiracleMix	10	29.44	5.30
II (2h)	MiracleMix	10	42.64	4.01
I (1w)	MiracleMix	10	48.12	4.34
II (1w)	MiracleMix	10	49.43	7.46
I (1w)	Fuji IX	10	52.70	6.57
II (1w)	Fuji IX	9	75.64	6.72
I (1w)	Vitremer	10	63.11	4.30
II (1w)	Vitremer	10	63.10	12.12
I (1w)	Z100	10	125.81	16.01
II (1w)	Z100	9	156.71	8.91
I (6w)	HiDense	8	63.86	11.53
II (6w)	HiDense	10	77.55	7.26

Condition* :The specimens of Condition I were stored in 100% relative humidity at room temperature. The specimens of Condition II were stored in water at room temperature.

N*: Number of the specimens

Table 4.7

The result of the statistical analysis of Preliminary Study Three (Two way ANOVA test).

Source	df*	SS**	F ratio	P value
Materials (A)	5	130231.03	356.10	<0.0001
Conditions (B)	1	6476.11	88.54	<0.0001
AB interaction	5	3763.21	10.29	<0.0001
Error	104	7607.06		

df*: Degree of freedom

SS**: Sum of Square

4.6.4 Discussion

The role of the water is very important for the maturation process of GICs (see Section 3.3.2). The effect of evaporation of water from GICs was reported by Wilson *et al.* (1979) who described the water as 'loosely bound' and 'tightly bound' water. Loosely bound water is evaporable and can easily be removed by desiccation. Tightly bound water is non-evaporable and cannot be removed by dehydration at room temperature. It is known that as the cements mature, they are more stable in terms of their water balance. When the cements dehydrate, they show deteriorated physical properties (Wilson and McLean, 1988). In this preliminary study, the specimens in a humidor showed significantly lower physical strengths compared to those immersed in water. They also showed some colour change while stored in a humidor. These results suggested that the condition of 100% relative humidity at room temperature was not successful to maintain the water balance of GICs and resulted in desiccation.

It was interesting to note that Z100 had also showed lower physical strength when stored in a humidor than when stored in water although no colour difference was observed. Compared to GICs, CR was expected to show minimum influence on desiccation due to its limited water content. Further research is needed to obtain a clearer understanding of this finding.

In *in vitro* studies, some researchers have adopted the 100% relative humidity storage condition instead of coating on the GIC specimen to control moisture prior to the strength tests (Mitra and Kedrowski, 1994; Suzuki *et al.*, 1995; Mount *et al.*, 1996). This has the advantage of preventing the water soluble ions washing out and has also been considered to maintain the water balance of GICs. Some researchers adopted the 100% humidity condition at 37 °C (Mitra and Kedrowski, 1994; Suzuki *et al.*, 1995) while some researchers used the 100% condition at room temperature (Mount *et al.*, 1994) prior to the strength tests.

In this experiment, the specimens were stored at room temperature, not at 37 °C. The degree of GIC dehydration in the atmosphere at higher temperatures such as at 37°C may not be a significant problem compared to the one at room temperature as air can accommodate more water per volume as the temperature rises. Wilson and Paddon (1993) studied conventional GICs in different conditions at 37 °C. In their study, the specimens were sealed for 20 minutes and then exposed to environments of 50% and 100% relative humidity and in water at 37 °C for 34 hours. They found that the contraction of GICs under the desiccating condition (50% relative humidity) was far greater (-0.56%) than the expansion by water absorption (+0.032% in water). The specimens under the 100% relative humidity condition showed the smallest dimensional change (+0.002%).

Further investigation should be carried out to determine the most suitable storage conditions for *in vitro* GIC tests in order to obtain more clinically relevant results. In that case, conditions involving saliva should be taken into account in further study.

4.6.5 Conclusion

Both GIC and CR specimens showed the tendency of reduced strengths when stored at 100% humidity at room temperature compared to those stored in water. This would suggest that the condition of 100% relative humidity at room temperature results in dehydration of the materials.

4.7 Preliminary Study Four-Light cure time and the shear strength

4.7.1 Introduction

It is generally well known that there is a limitation to the depth of cure in light-cured materials. However, it is sometimes confusing as each manufacturer describes a different light cure requirement. Many manufactures simply supply the information based on one standard shade, while some manufactures describe several variations of the time and the depth of several common shades. For example, in the case of Vitremer, it was specified that the depth of cure was optimal for 40 seconds irradiation as 2.5 mm for A-3 shade and 2.0 mm for A-4 shade.

As there were no previous data describing the effect of the light exposure time on the shear strength of the 1.0 mm thick shear punch materials, the shear punch strengths of different light exposure time to specimens was examined in this part of the study.

4.7.2 Materials and methods

Two materials, Pertac Hybrid and Vitremer were used in this experiment. The shades of the two materials were both A3. According to the manufacturers' information, a 40 second light exposure was necessary for curing a 2 mm thickness of both Pertac Hybrid and Vitremer specimens. A total number of forty specimens were prepared in the same manner previously described (see Section 4.3.2), except for the following procedures. Ten specimens of each material were light cured for 20 seconds (Group I), and another ten specimens of each materials were light cured for 60 seconds (Group II). Immediately after light curing, the specimens were all stored in water and kept in the dark to keep out further light exposure for one week and then the shear strength was tested. Differences between the materials and the curing times were assessed by two way ANOVA tests.

4.7.3 Results

The results of this preliminary study are shown in Figure 4.9, Table 4.8 and Table 4.9. These results suggest that there were no significant differences between the specimens cured for 20 seconds and 60 seconds ($p > 0.05$). Pertac Hybrid showed slightly higher strengths with longer curing while the Vitremer specimens showed similar strengths in both groups.

Table 4.8

The results of the shear punch tests of Preliminary Study Four. The specimen size, the mean strengths and the standard deviations of the specimens.

Group*	Materials	N**	Mean strength (MPa)	SD
I	Pertac Hybrid	10	134.37	11.12
II	Pertac Hybrid	10	145.92	8.87
I	Vitremer	10	64.29	8.37
II	Vitremer	10	63.42	8.82

Group*: The specimens of Group I were light cured for 20 seconds. The specimens of Group II were light cured for 60 seconds.

N*: Number of the specimens

Table 4.9

The statistical analysis of the results of Preliminary Study Four. (Two way ANOVA test)

Source	df*	SS**	F test	P value
Material (A)	1	58197.06	620.79	0.0001
Condition (B)	1	285.58	3.05	0.0895
AB	1	385.02	4.11	0.0502
Error	36	3374.905		

df*: Degree of freedom

SS**: Sum of Square

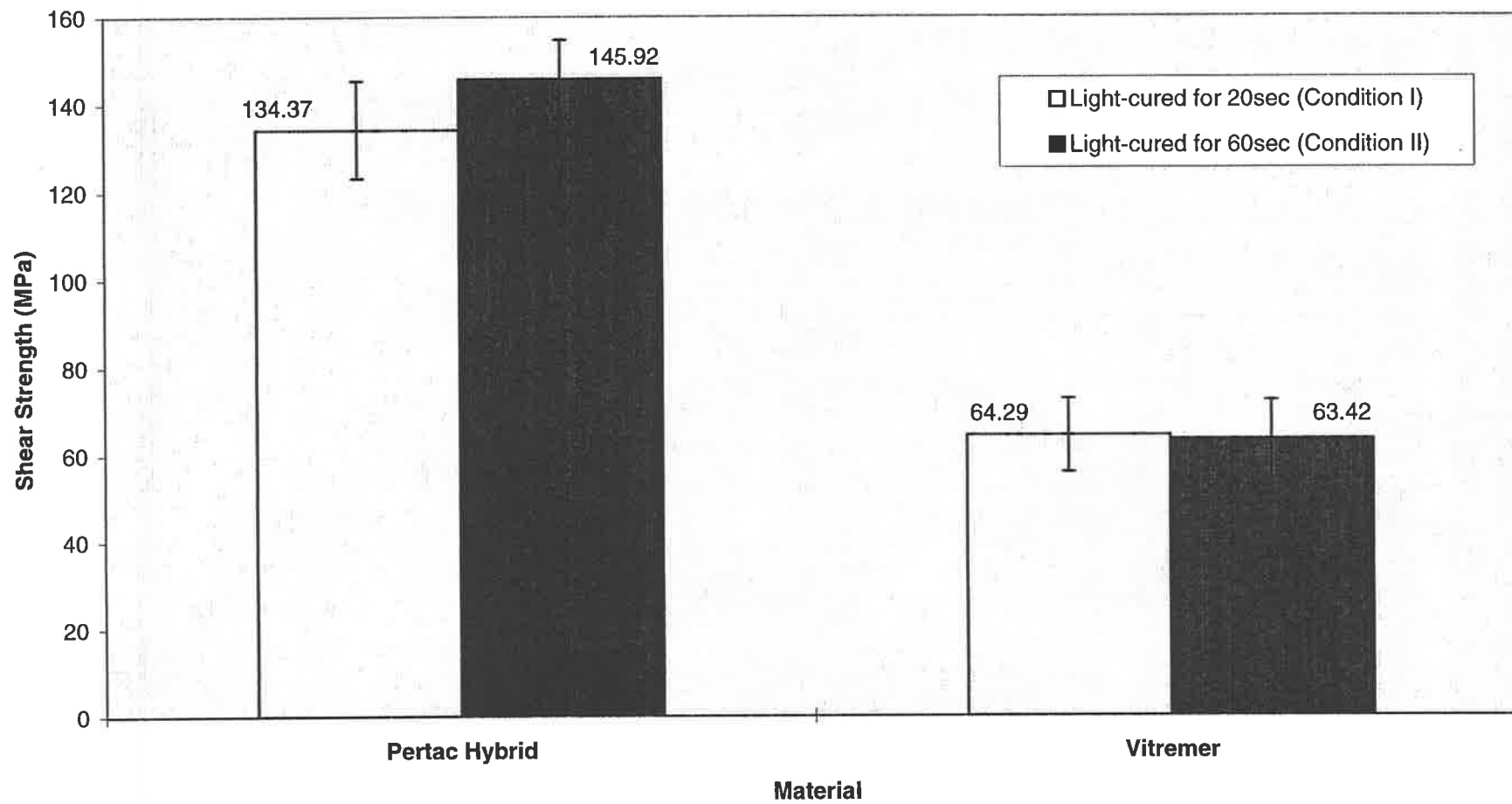


Figure 4.9

Preliminary Study Four Light Cure Time and Shear Punch Strength

The mean shear strength of two light-cured materials stored in water for one week after curing.

* Error bars represent the standard deviation.

4.7.4 Discussion

RM GICs can be set in darkness (McLean *et al.* , 1994), and the manufacturers claim that the on-going acid-base reaction would compensate for the disadvantages, such as the limitation of the depth of cure, which are found in other light cure materials. Also, they claim that the snap setting of the cements due to the light activation reduced the sensitivity to moisture so that it is possible to polish the restorations in the same day. Some questions arise about these claims. Firstly how much of the material would be cured with the light activation and how much will be left for the 'on-going' acid-base reaction? Secondly, what sort of correlation is there between the depth of cure and the light exposure time? The situation is now confusing because the manufactures' information regarding the light exposure time and the depth of cure of RM GICs and CRs are not standardized. Most manufacturers showed times necessary for obtaining a depth of cure of 2.0 -3.0 mm which varied between 20- 40 seconds.

In this study, the Pertac Hybrid specimens with 20 second light exposure time showed lower strengths than those with 60 second light exposure time although the results of the statistical analysis showed no significant differences between the two groups ($p>0.05$). On the other hand, the Vitremer specimens showed very similar strengths in the two groups. The difference of the strengths found in the two Pertac Hybrid groups may merely have resulted from an experimental error, but it may reflect the completion of the cure. Considering that 40 seconds was the recommended curing time for specimens of 2 mm thickness, the 1 mm thick specimens with the exposure of the light for 60 seconds was considered as complete cure of the light activated component compared to those with the 20 second exposure time. The very similar strengths found in the two Vitremer groups may be the result of the acid-base reaction over the one week storage period.

Based on this study, the final protocol was established with exposure of the shear punch specimens to the light for 40 seconds to ensure complete cure. It should be noted that the specimens with a longer light exposure time were at the same time sealed longer with mylar strips which controlled the moisture balance longer. Meanwhile the auto-cure component of the material would proceed and the water sensitivity might be decreased resulting in higher strengths. These several factors appear to compound and influence over all properties.

4.7.5 Conclusion

The light exposure time of 40 seconds was adopted as the final protocol for all the light activated materials.

4.8 Preliminary Study Five - Maturation of GICs and CRs

4.8.1 Introduction

It appears to be generally accepted that GICs exhibit an increase in strength over a long period of time (Crisp *et al.*, 1976a; Suzuki *et al.*, 1995). Some researchers have stated that GICs reach an initial peak strength after 24 hours from mixing (Pearson and Atkinson, 1991; Williams and Billington, 1991; Cattani-Lorente *et al.*, 1993), while some literature indicates that even after half year, cure may still be proceeding (Crisp and Wilson, 1974). This slow curing of GIC is often so called 'maturation'. However, some studies have shown the reduction of the GIC strengths over time (Pearson and Atkinson, 1991; Williams and Billington, 1991; Soltesz and Leupolz, 1992).

Although little data on RM GICs have been reported, there is evidence that the curing process of RM GICs may last 24 hours or more (Yoshikawa *et al.*, 1994; Swift *et al.*, 1995, Uno *et al.*, 1996). In this study, the strengths of GICs stored in water for 2 hours and 1 week at room temperature were compared and the effect of maturation over time was investigated.

4.8.2 Materials and methods

Two groups each of ten specimens of Z100, Pertac hybrid, Fuji II LC Blue and Miracle Mix were prepared in the manner described previously. Groups of specimens were stored in water for 2 hours and for one week at room temperature, respectively. The shear strengths were measured and compared by a two way ANOVA test.

4.8.3 Results

The result was shown in Figure 4.10, Table 4.10 and Table 4.11.

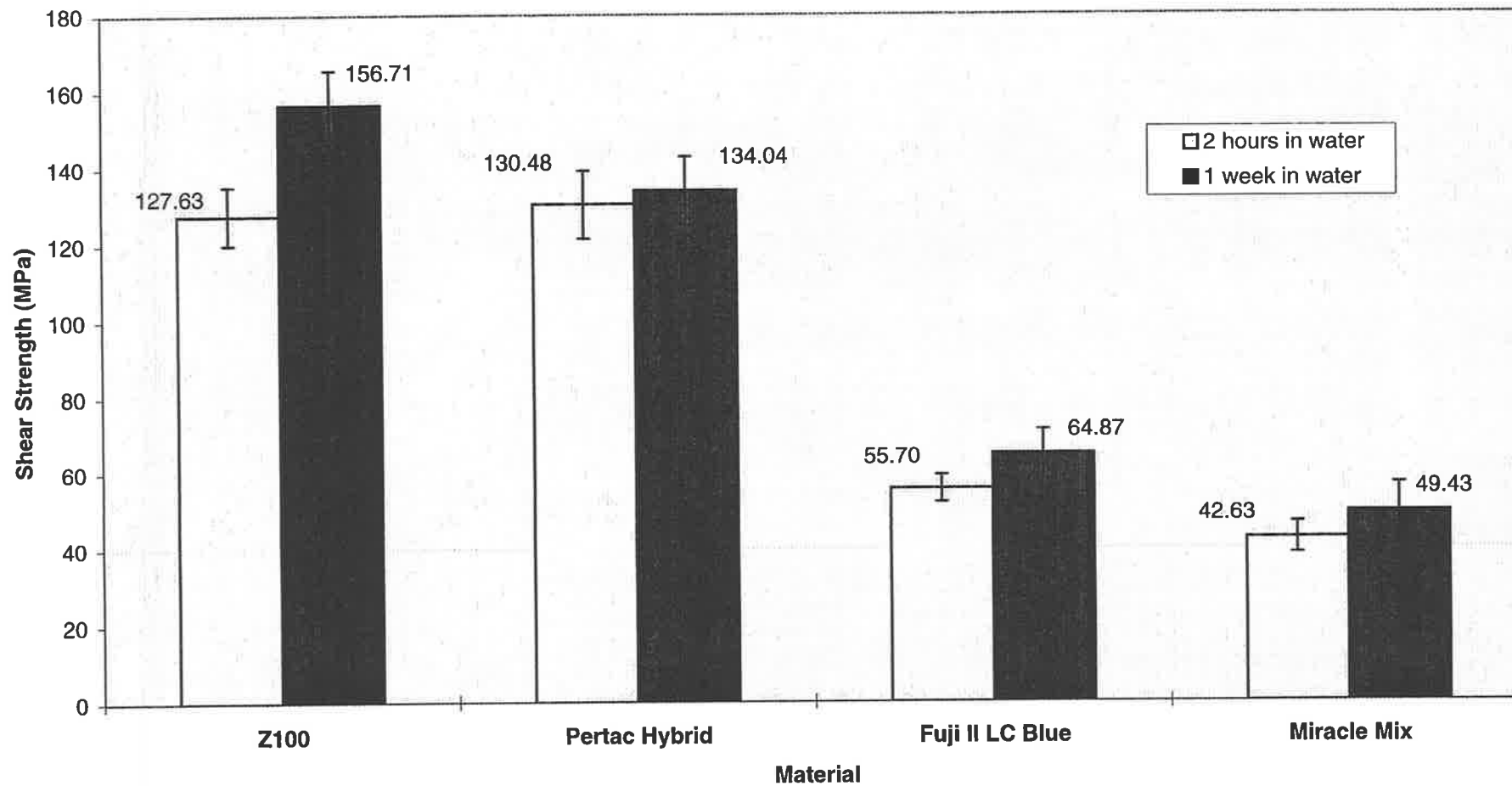


Figure 4.10

Preliminary Study Five *Maturation of GICs and CRs*

The mean shear strength of two GICs and two CR stored in water for 2 hours and for one week.

* Error bars represent the standard deviation.

Table 4.10

The results of the shear punch tests of Preliminary Study Five. The sample sizes, the mean strengths and the standard deviations of the specimens.

Group*	Materials	N**	Mean strength (MPa)	SD
I	Z 100	10	127.63	7.59
II	Z 100	10	156.71	8.91
I	Pertac Hybrid	10	130.48	8.87
II	Pertac Hybrid	9	134.04	13.26
I	Fuji II LC Blue	10	55.70	3.60
II	Fuji II LC Blue	9	64.87	6.53
I	MiracleMix	10	42.63	4.01
II	MiracleMix	10	49.43	7.46

Group*: The specimens of Group II were tested after 2 hours storage in water. Those of Group I were tested after one week in water.

N*: Number of the specimens

Table 4.11

The results of the statistical analysis of Preliminary Study Five. (Two way ANOVA test)

Source	df*	SS**	F ratio	P value
Materials (A)	3	139197.00	722.148	<0.0001
Groups (B)	1	2826.71	434.00	<0.0001
AB	3	1894.17	9.827	<0.0001
Error	69	4433.34		

df*: Degree of freedom

SS**: Sum of Square

4.8.4 Discussion

Slow setting characteristics of GICs have been reported by many researchers (Wilson and McLean, 1988; Wilson and Nicholson, 1993; Mount, 1994b), although the setting reaction of GICs is very complicated and not fully understood (Crisp *et al.*, 1976b; Pearson and Atkinson, 1991). In this study, all the tested materials (both GICs and CRs) tended to show an increase in strength over time. A similar trend was shown by Mount *et al.* (1996) who compared GICs and CRs after 2 hours and 5 days. It was interesting to see Z100 showed a more noticeable increase in strength after one week than the other materials despite the fact that no acid-base reaction was taking place in CRs. Several researchers have reported that CRs activated with the light show increased strength over time even after light activation (Watts *et al.*, 1986; Pilo and Cardash, 1992). It has also been found that the free-radical polymerization reaction initiated by visible light exposure continues after cessation of the light exposure (Pilo and Cardash, 1992). This kind of 'on-going' reaction was often described as a disadvantage for those materials as it may be accompanied by polymerization shrinkage. There are relatively few studies of curing shrinkage of conventional GICs (Feilzer *et al.*, 1988; Hinoura *et al.*, 1992, 1993; Lai and Johnson, 1993), and studies of curing shrinkage of RM GICs are rare (Attin *et al.*, 1995, Feilzer *et al.*, 1995). Further studies in this area should be encouraged.

4.8.5 Conclusion

In this experiment, both GIC and CR shear punch specimens stored in water for one week showed higher strengths than those stored for two hours. Many studies of the physical properties of GICs have been carried out after 24 hours from mixing, however one week storage time was selected for the subsequent experiments to allow both GICs and CRs more chemical maturation.

4.9 Discussion

Throughout the series of the preliminary studies, the standard deviation of the results were small (mostly 5-10% of the mean values), which suggested that the results of the shear punch test were reproducible. Furthermore, the most frequently adapted sample size (N=10) has shown to be sufficient to demonstrate significant differences ($p < 0.05$) by a one-way and two-way ANOVA tests.

In testing GICs, the treatments of the specimens and the procedures of the experiments are important. Several points of the final protocol were determined based on the information from the series of pilot studies in this chapter. The sample size of each preliminary study was too small to make any wide ranging conclusions, however several important factors influencing the

results of the shear punch tests were addressed through these studies. In particular, the influence of water should be taken into account.

The GIC specimens used in this study were not coated. This decision was made for several reasons. Firstly, coating added a number of complicating factors. It was difficult to see if the coating materials were completely removed from the specimens when they were ground prior to punching. Also, the specimens were very thin and the influence of the extra thickness of the coating material was considered to be greater than the influence of the water contamination. Based on the result of the preliminary study, no significant difference was found in the strengths between the coated and uncoated specimens. Further, in clinical terms, it is often difficult to achieve perfect coating in the oral environment due to factors such as occlusion, food and the movement of the soft tissues such as the cheek and tongue, thus there is clinically a high possibility that restorations are exposed in water a relatively short time after mixing. The usage of a humidifier was the other possible solution, however, the preliminary study results showed it was not appropriate. Therefore, all the specimens were exposed to water after initial setting.

Although some experimental limitations still exist in this study, more controlled shear punch results were expected using the protocol developed from the data obtained from these preliminary studies.

4.10 Conclusion

These tests provided preliminary data for comparison of materials and allowed examination of the effects of manipulation. Based on these preliminary studies, the following protocol was established to test GICs and CRs:

- 1) GIC specimens are not coated.
- 2) after the initial set, the specimens are stored in water for one week at room temperature.
- 3) all light cure materials are cured for 40 seconds.
- 4) 10 specimens are made for each group.

Apart from these changes, the same protocol described in this chapter (Section 4.3.2) was adopted for subsequent studies.

Chapter 5

Powder/liquid Ratio and the shear strength

5.1 Overview

The influence of the powder/liquid ratio (P/L) has often been discussed as a significant factor in the strength of GICs in the literature (Mount, 1994b). In this chapter, as part of the preliminary study of the shear punch test, the influence of P/L was investigated in the following experiments:

- 1) Preliminary Study Six: Powder/liquid ratio of dispensed GICs
- 2) Preliminary Study Seven: Powder/liquid ratio and the shear strength

5.2 Introduction

In past, several possible modifications were employed to improve the physical properties of GICs. The following five factors were suggested by Wilson and McLean (1988) as influencing the physical strength of GICs:

- variation in glass powder
- powder/liquid ratio
- hydration or dehydration of the cement
- porosity
- molecular weight of the liquid

Billington *et al.* (1990) in an interesting study of P/L in GIC restorative cements (ChemFil II, Dentsply) mixed by 22 dental surgery assistants found that all mixes contained less powder (mean ratio 5.0:1) than the manufacturer's recommended ratio (6.5:1). Ratios varied greatly among assistants, but mixes by the same assistant were generally similar to each other, even though most participants in this study did not use the supplied measures when mixing. Specimens mixed with the mean ratio described in this study showed compressive and diametral strengths were about half of the strengths of specimens prepared with recommended P/L.

It is generally accepted in conventional GICs, compressive strengths increase as the P/L is increased (Crisp *et al.*, 1976a). The newly developed RM GICs have been assumed to be similarly sensitive to variation in P/L although there is little data reported in the literature. The

aim of this chapter was to investigate the influence of P/L on the shear strength in conventional and the RM GICs.

5.3 Preliminary Study Six - Powder/liquid ratio of dispensed GICs

5.3.1 Introduction

The way of dispensing GICs are usually prescribed by manufacturers. In traditional powder-liquid type GICs, the methods involve the use of supplied measuring spoons and corresponding drops from the liquid bottle. The methods, however, vary from manufacturer to manufacturer. According to Tsuchitani (1993), essential points for proper measurement are:

- shaking the bottle of the powder before dispensing the powder,
- closing the lid immediately after dispensing, not putting the left-over powder back into the bottle,
- for the liquid, dropping the drops vertically from above the mixing pad.

Also Mount (1994b) described the method of dispensing GICs in detail. In this study, several powder-liquid type GICs were tested to investigate how much variation was observed in dispensing the liquid and powder.

5.3.2 Materials and methods

Four powder-liquid type GIC materials were used in this study. The description and details of these materials are listed in Table 5.1

Table 5.1

Four powder-liquid type GICs and their details used in the experiments in Chapter 5

Products	Manufacturer	Batch #	Description
Fuji I	GC Co., Tokyo Japan	021051	C GIC
Fuji IX	GC Co., Tokyo Japan	110341	C GIC
Fuji II LC	GC Co., Tokyo Japan	liquid/070731 powder/100835	RM GIC
Vitremer	3M Dental, St. Paul, USA	19930223	RM GIC

RM GIC: Resin-modified GIC

C GIC: Conventional GIC

For each material, the number of drops of the liquid was determined to ensure approximately 0.75g of mixture which was the maximum volume which a tip of the Centrix syringe (Centrix Inc, Shelton, USA) was able to hold. Liquid was dispensed by holding the liquid vial vertically with the dropper tip down without the tip contacting the mixing pad and the bottle was squeezed to dispense drops onto the measured glass slab. The weight of liquid was carefully measured by an electronic balance (AC-400, Phoenix, Denver, USA).

The powder jar was shaken to fluff the powder before dispensing. The attached spoon was inserted into the jar, and overfilled with loosely packed powder and the appropriate number of scoops corresponding to the number of the drops of liquid were leveled. Then each P/L was calculated and compared with standard P/L. This procedure was repeated 6 times for each material.

5.3.3 Results

Table 5.2 shows the results of the weights of the dispensed powder and liquid and P/Ls of the four GICs. The mean P/Ls of the dispensed GICs were 1.72:1 (Fuji I), 3.11:1 (Fuji II LC), 3.87:1 (Fuji IX) and 2.64:1 (Vitremer) while the ratios recommended by the manufacturers were 1.8:1, 3:1, 3.6:1 and 2.5:1, respectively.

Table 5.2

The variations of the weights of liquid and powder (g) and P/L of the four GICs

Materials	Weight of the liquid (g)	Weight of the powder (g)	P/L of the mixing cement	Percentage of the powder*
Fuji I	<u>4 drops</u>	<u>2 scoops</u>		
	0.251	0.473	1.88:1	5% ↑
	0.257	0.456	1.77:1	1% ↓
	0.264	0.448	1.69:1	6% ↓
	0.278	0.451	1.62:1	10% ↑
	0.266	0.451	1.69:1	6% ↓
	0.270	0.448	1.66:1	8% ↓
Fuji H LC	<u>6 drops</u>	<u>3 scoops</u>		
	0.195	0.610	3.12:1	4% ↑
	0.127	0.417	3.28:1	9% ↑
	0.125	0.391	3.13:1	4% ↑
	0.134	0.434	3.24:1	8% ↑
	0.135	0.392	2.90:1	3% ↓
	0.121	0.360	2.98:1	1% ↓
Fuji IX	<u>3 drops</u>	<u>3 scoops</u>		
	0.170	0.671	3.95:1	10% ↑
	0.173	0.678	3.92:1	9% ↑
	0.175	0.672	3.84:1	8% ↑
	0.180	0.661	3.67:1	2% ↑
	0.187	0.723	3.87:1	12% ↑
	0.180	0.709	3.94:1	9% ↑
Vitremer	<u>4 drops</u>	<u>4 scoops</u>		
	0.214	0.567	2.65:1	6% ↑
	0.208	0.557	2.68:1	7% ↑
	0.185	0.542	2.93:1	6% ↑
	0.195	0.517	2.65:1	7% ↑
	0.203	0.543	2.67:1	10% ↑
	0.214	0.484	2.26:1	5% ↓

* The percentage of the excess (↑) or less (↓) powder of the mixture when the weight of the scooped powder was compared with those which calculated from the weight of the dispensed liquid based on the manufacturers' recommended P/L.

5.3.4 Discussion

It is known that errors in the P/L for cement type materials affect the properties of the cement (Saito, 1993; Wilson and Nicholson, 1993; Mount, 1994b). For GICs, this is largely related to the water-sensitive period, significantly affecting overall physical properties and clinical results (Saito, 1993).

Although this experiment was carefully conducted, there were approximately 5-10% average differences between the recommended P/Ls and those found in the dispensed materials. Several researchers have reported a range of the differences in the different systems. Braem *et al.* (1995) studied restrained fracture strength of both hand-mixed and capsulated Fuji II LC, and reported that a scoop of powder and droplets of liquid of the powder-liquid type could range from 50 to 150% of the average weight, depending on the way these were dispensed. Mount (1984) had shown that the ChemFil system dispensed 2% below the standard P/L while Fuji II showed 8% difference between the manufacturer's recommended P/L.

Two independent factors could contribute to these differences. Firstly, the weight of each drop of the liquid varied. Secondly the weight of the each scoop of the powder varied. Moreover, the presentation of the powder and the liquid varied from product to product. The smaller bottles such as Fuji IX and Vitremer were difficult to scoop the powder from, as they presented very narrow outlets. Perhaps this resulted in more condensed powder when scooped and these products showed the tendency of the higher P/Ls compared with the manufacturers' recommended P/Ls.

Not surprisingly, the instructions of each product regarding the mixing methods varied. Among the tested GICs, the instruction for Vitremer (3M Dental, St. Paul, USA) appeared to be the closest to the recommendation found in the literature. Some instructions of the tested materials did not always provide sufficient information for consumers to ensure correct handling. For example, it is known that the first couple of drops from a newly opened liquid bottle are smaller than drops dispensed later (Saito, 1993). All the liquid bottles used in this study had already dispensed several times prior to this experiment so that the results did not record the weight of the newly dispensed drops. However, in the preliminary study, it was observed that the few drops dispensed from the newly opened liquid bottles were smaller as Saito (1993) described. Obviously the mixture made from these drops would show a stiffness which could cause difficulty in handling in the clinical situation. Such information should be included in the manufacturer's instructions, but none of the instructions of the tested materials described it clearly. Clinicians should be aware that the dispensing systems for each material are not the same. Also, more careful handling in dispensing should be emphasized in the clinical situation

for both clinicians and dental staff. In spite of very conscious efforts, the present study still showed a relatively high variation.

Due to the limitation to control of the P/L, capsulated types can be recommended. It is assumed that mixed GICs with the capsulated system show more constant properties, as their P/L is controlled during manufacturing, and they are mixed automatically. In this context, Mount (1994b) suggested that clinicians should be aware of the variation in trituration machines in each clinic, as it can affect working time and physical properties.

5.3.5 Conclusion

The P/Ls of the dispensed materials showed approximately 5-10% variation compared to the recommended P/L even under controlled condition such as in this study. In the clinical situation, it is important for the persons who dispense and mix GICs (mainly dental auxiliaries) to be aware of the need for a standardised method and the effects of the different P/Ls on GICs.

5.4 Preliminary Study Seven - Powder/liquid ratio and the shear strength

5.4.1 Introduction

It is generally known that the strength of GICs increase as P/L increases (Crisp *et al.*, 1976a). However, this may be different between conventional and resin-modified GICs. In this study, the shear punch strengths of specimens prepared with different P/Ls were investigated.

5.4.2 Materials and methods

The details of the two GIC materials used in this study is described in Table 5.3. Both were the powder/liquid type. A total of ninety specimens were made. Six groups of Fuji I specimens and three groups of Vitremer specimens were prepared in the manner described in Chapter 4. On each occasion when dispensing the materials, the weights of the liquid were first measured by an electric scale (AC-400, Phoenix, Denver, USA), and according to this weight, the appropriate weight of the powder was measured and mixed. Specimens were stored in water at room temperature for one week, and the shear strength was investigated.

Table 5.3

Two GICs and their details used in this study

Products	Manufacturer	Batch #	Description	Recommended P/L
Fuji I	GC Co., Tokyo Japan	021051	C GIC	1.8:1
Vitremer	3M Dental, St. Paul, USA	19930223	RM GIC	2.5:1

C GIC: Conventional GIC

RM GIC: Resin-modified GIC

Table 5.4

The results of the shear punch tests of the two kinds of GICs with the variety of the P/Ls.
The sample sizes, mean strengths and the standard deviations of the specimens.

P/L	Percentage of powder**	Materials	N**	Mean strength (MPa)	SD
1.4:1	22%↓	Fuji I	10	53.55	6.97
1.6:1	11%↓	Fuji I	10	57.37	4.88
1.8:1*	—	Fuji I	8	60.15	6.68
2.0:1	11%↑	Fuji I	10	61.45	5.64
2.2:1	22%↑	Fuji I	10	60.98	4.22
2.5:1	38%↑	Fuji I	10	62.74	6.24
2:1	20%↓	Vitremer	10	55.91	6.75
2.5:1*	—	Vitremer	10	63.10	12.12
3:1	20%↑	Vitremer	10	65.30	4.86

* Manufacturer's recommended P/L

** The percentage of the excess (↑) or less (↓) powder of the mixture when the weight of the scooped powder was compared with those which calculated from the weight of the dispensed liquid based on the manufacturers' recommended P/L

N*** Number of the specimens

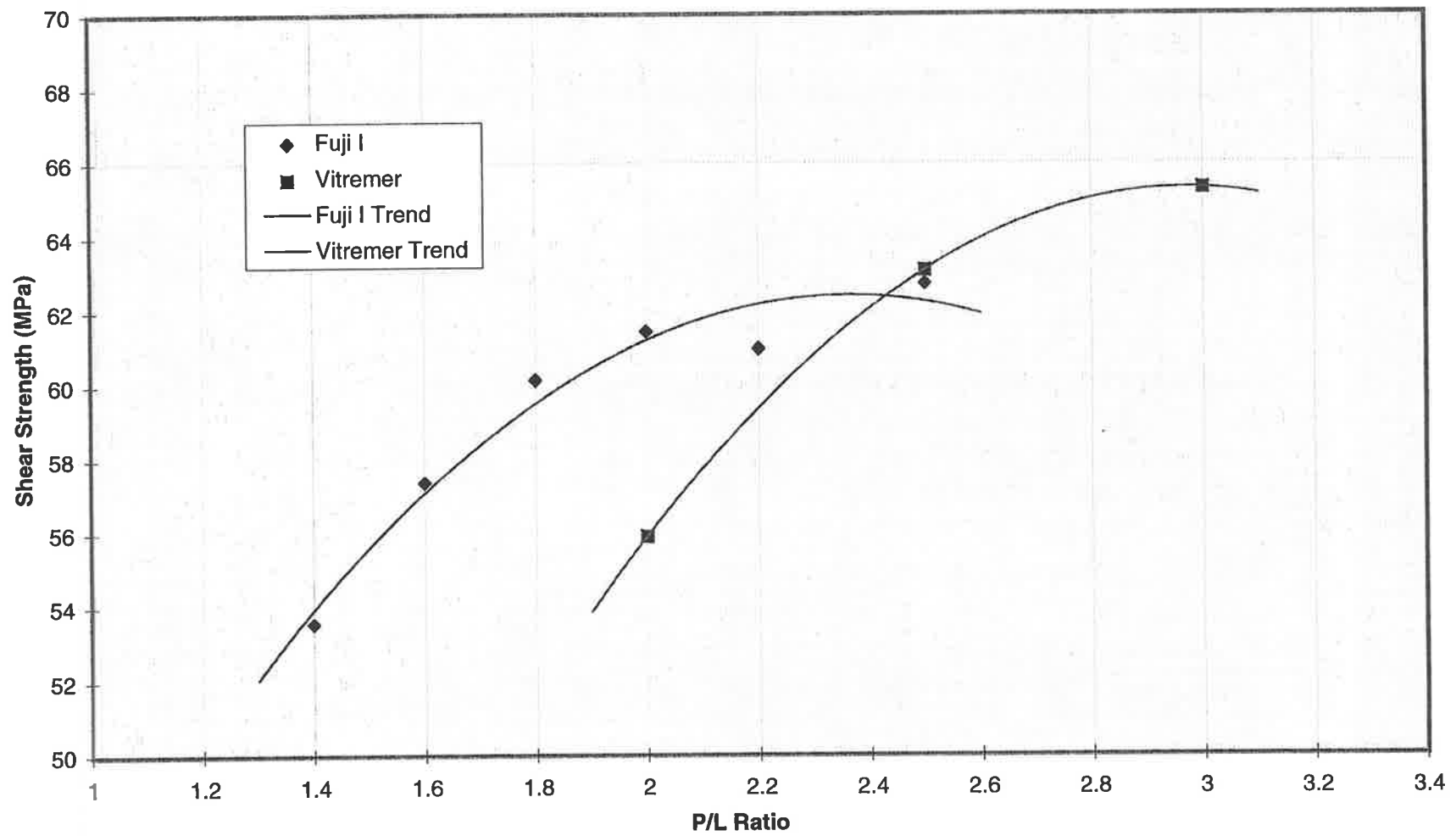


Figure 5.1
 Preliminary Study Seven *Variation of Mean Shear Strength of Fuji I and Vitremer with P/L Ratio*

5.4.3 Results

The results are shown in Figure 5.1 and Table 5.4. It was found that strength increased as the P/L increased. This tendency was found in both the resin-modified and the conventional GICs. This increase was observed as the P/L increased up to the recommended ratio. At greater P/L, the rate of increase in strength was smaller than the rate of decrease in strength below the recommended ratio.

5.4.4 Discussion

As previously reported, both the tested materials showed an increase in the strength as the P/L increased. However, the variation in the strengths of this study was relatively small compared to those reported by Crisp *et al.* (1976a) where the rate of the reduction in the strength showed much more rapid change with the reduction of the powder. For example, in their study, a 10% reduction in the powder content resulted in a loss of at least 10% in the compressive strength while in this study the same amount of reduction in the strength was found when the powder was reduced approximately 20%. These differences may have resulted from usage of the different materials, the experimental conditions and the nature of the strength tests. Saito (1993) studied the relationship of the P/L, the compressive strength and setting time of the GIC. They also found that the material showed lower strength when the more liquid was used. Similar to the results by Crisp *et al.* (1976a), they reported approximately 10% reduction in strength in the GIC which powder component was reduced 10% from the manufacturer's recommended ratio.

One question which arises is about what the 'recommended P/L' means. Obviously, the manufacturers decide it based on the data which they independently collect and determine the P/L at which the material would perform the best. In fact it was difficult to justify the manufacturer's recommended P/L from this data alone. Firstly, as seen in Figure 5.1, the increase in strength continued even in greater P/L than the manufacturer's recommended P/L, although the rate of the increase in strength was reducing as the ratio increased. Secondly, in this experiment, the mixture of the 2.5:1 P/L Fuji I (approximately 40% more powder) was found to be too viscous but the cements in the other ratios still appeared to give an acceptable handling character. The mixture of 3:1 P/L Vitremer (20% more powder) was observed to be too opaque for aesthetic restorative purposes, but showed acceptable workability.

Crisp *et al.* (1976a) concluded that GICs (for restorative purposes) should be mixed with the highest P/L possible to produce cement pastes which have rapid hardening rates, high strengths

and the greatest resistance to the effect of moisture. This suggestion may be beneficial from the aspect of physical properties, but, there is a limitation to this procedure as there reaches a point where translucency declines due to unreacted particles and the clinical workability is not acceptable (Mount, 1994b).

Consequently, it is difficult to determine the best P/L to balance these complex factors. Moreover, a broad range of properties and the applications of each material should be taken into consideration. The strength is just one of the criteria to consider. In this study, for example, the two tested materials should be assessed differently. For example, besides the physical properties, luting cements such as Fuji I should meet criteria such as film thickness. Aesthetic restorative materials such as Vitremer should be assessed by color and translucency.

5.4.5 Conclusion

Both conventional and resin-modified GICs showed increase in strengths as the P/L was increased although the increase in strength at P/L greater than the recommended ratio was small. The definition of the 'recommended P/L' should be clarified and the efficacy of these should be further assessed.

5.5 Discussion

It is important to be aware that the constituents of the various GICs currently available are not the same. The size of the powder particles, the formula of the powder and the liquid, viscosity, and working time are all different (see Section 3.3). Naturally, those handling these materials need a good understanding of each product. However, it is often confusing because of the limited information available especially when the material is newly introduced.

It also confusing that manufacturers supply a variety of standard P/L which vary between products. For example, powder/water type GICs generally appeared to be mixed with higher P/Ls. These types of GICs involve the glass powder blended with 'dried' polyalkenoic acid. Examples of those products are ChemFil II™ (Dentsply, West Germany) and Legend™ (S.S.White Manufacturing, England). The recommended standard ratios were 6.8:1 and 7.0:1, respectively. On the other hand, powder-acid type GICs generally employed lower P/Ls of the order of 3.2:1 for KetacFil™ (ESPE, West Germany), and 2.5:1 for Vitremer™ (3M, USA).

The results of this study suggested that the cements with a higher P/L showed higher strength within the same material. However, the GICs with high P/L such as ChemFil II and Legend do not necessarily show superior strength to those with low recommended P/Ls. For instance,

Cattani-Lorente *et al.*, (1993) reported that Fuji II (P/L 2.3:1) showed higher flexural and tensile strengths than ChemFil II (P/L 6.8:1) while the compressive strength showed the opposite result. The other argument is how to set up the 'recommended P/L'. As previously discussed, there are many factors involved in this, and the standard methods to determine those issues should be addressed in future.

The current dispensing systems adopted in powder/liquid type GICs obviously need further improvement. It is important to control the P/L as the variation in the P/L affects the quality of the restorations. Based on the results from these preliminary studies, all the powder/liquid type GICs used in this study were mixed with the manufacturers' recommended P/L using an electronic scale.

5.6 Conclusion

Care must be taken in handling GICs because mixing with a lower P/L results in weakened physical properties in both conventional and resin-modified GICs. Clinicians should be aware of the differences of their consistency and handling of each GIC as there are several different systems even within the conventional GICs. Proper training and education of dental staff is also mandatory.

The capsulated type of GICs may be recommended as a 5-10% variation of P/L in dispensing powder/liquid type GICs was inevitable even under the controlled dispensing methods.

Chapter 6

Comparison of the Strength of GICs and CRs

6.1 Overview

As previously discussed, the shear punch tests adopted in this study are not the standard ISO tests and limited comparative data are available in the literature. The shear punch strength data of commonly available GICs and CRs were collected to allow a comparison between materials.

6.2 Introduction

The applications of GIC restorative materials are relatively limited compared to CR restorative materials mainly because of their lower physical strengths. Recently developed RM GICs were reported to have improved properties (Mathis and Ferracane, 1989; Mitra, 1991; Kitamura *et al.*, 1993; Nathanson and Butthieu, 1993; Tosaki and Hirota, 1994) although the information of RM GICs were still limited due to their relatively short history.

The strengths of GICs and CRs were investigated in various ways by various researchers (McCrary and Powers, 1992, 1993; Willems *et al.*, 1992; Cattani-Lorente *et al.*, 1993; Hammesfar, 1994; Mitra and Kedrowski, 1994; Suzuki *et al.*, 1995; Uno *et al.*, 1996). ISO describes different standardized tests for each material (for CRs; the flexural test, ISO4049:1988; for GICs; the compression test, ISO 9917:1991) and the results from these two different tests make it difficult to compare the materials. In addition, the special precautions for handling GICs during the experimental process (such as avoiding dehydration) should be addressed or the data for GICs derived from the test may be artificially low. In this study, the strength of a broad range of currently available CRs and GICs including some RM GICs were compared using the shear punch test.

6.3 Materials and methods

The details of the materials (7 GICs and 5 CRs) studied in this chapter are described in Table 6.1. Among the GICs listed, HiDense and Miracle Mix were capsulated versions, otherwise all were powder-liquid type versions. Fuji II LC, Fuji II LC Blue and Vitremer are resin-modified GICs and both Miracle Mix and HiDense are GICs with the inclusion of a metal component in the powder. Among CRs, Degufill M and Silux Plus were microfilled type CRs while the others were hybrid type CRs. All CRs were light-cured materials. The materials were all widely available in Australia except for Fuji II LC Blue which was a trial material from GC Corporation (Tokyo Japan).

Unless specifically described, the methods described in Chapter 4 were adopted for the shear punch test in this chapter. The strengths of each material were compared and a one way ANOVA test using SPSS version 4.0 (Macintosh) was carried out. The materials were then divided into two groups (the GIC group and CR group), and the differences in the strengths between these two groups were assessed and a two way ANOVA test was carried out using SPSS version 4.0 (Macintosh).

Table 6.1

Manufacturer's details of the seven GICs and five CRs used for the shear punch tests in this chapter

Products	Manufacturer	Batch #	Description
Composite resins			
Silux Plus	3M Dental, St. Paul, USA	19940202	M CR
Z100	3M Dental, St. Paul, USA	19940222	H CR
Pertac Hybrid	ESPE, Seefeld, Germany	0067X301	H CR
Degufill H	Degussa, Frankfort am Main, Germany	119A2	H CR
Degufil M	Degussa, Frankfort am Main, Germany	116A2	M CR
GICs			
Fuji II LC Blue	GC Co., Tokyo Japan	050441	RM GIC/powder-liquid
Fuji II LC	GC Co., Tokyo Japan	liquid/070731 powder/100835	RM GIC/powder-liquid
Vitremer	3M Dental, St. Paul, USA	19930223	RM GIC/powder-liquid
Miracle Mix	Shofu, Kyoto Japan	920114	C GIC/capsulated
HiDense	Shofu, Kyoto Japan	931027-1	C GIC/capsulated
Fuji IX	GC Co., Tokyo Japan	110341	C GIC/powder-liquid
Fuji II Cap	GC Co., Tokyo Japan	300391	C GIC/capsulated

C GIC: Conventional GIC

RM GIC: Resin-modified GIC

H CR: Hybrid composite resin

M CR: Microfilled composite resin

6.4 Results

The result of this study is shown in Fig 6.1, Table 6.2 and Table 6.3 and the results of the statistical analysis are shown in Table 6.4 and Table 6.5. There were significant differences between the materials ($p < 0.05$) (Table 6.5). A comparison of the data for the GIC group and the CR group are shown in Table 6.3 and Table 6.4. The tested material showed considerable variation between the GIC and CR groups although none of GICs showed higher strength than any CRs. There were significant differences between the CR group and the GIC group ($p < 0.05$). Of all tested materials, Z-100 showed the highest shear punch strength and Miracle Mix the lowest. Within the CR group, Degufill M showed the lowest shear punch strength (approximately 1/3 of the strength of Z-100). Among the tested GICs, HiDense showed the highest value. Based on their shear punch strengths, the ranking of these materials was:

Z-100 > Pertac Hybrid > Silux Plus > Degufill H > Degufill M > HiDense > Fuji IX > Fuji II Cap > Fuji II LC > Vitremer > Fuji II LC Blue > Miracle Mix

Table 6.2

The results of the shear punch tests of this study. The description of the materials, the sample size, the mean shear punch strengths and the standard deviations of each tested material.

Materials		N*	Mean strength (MPa)	SD
M CR	Silux Plus	10	119.91	5.21
H CR	Z 100	9	156.71	8.92
H CR	Pertac Hybrid	9	134.04	13.26
H CR	Degufill H	10	106.30	11.67
M CR	Degufill M	10	94.42	6.98
RM GIC	Fuji II LC Blue	9	64.87	6.88
RM GIC	Fuji II LC	9	66.34	5.63
RM GIC	Vitremer	9	66.07	8.37
C GIC	MiracleMix	10	49.43	7.47
C GIC	HiDense	10	85.53	8.44
C GIC	Fuji IX	10	75.73	6.72
C GIC	Fuji II Cap	10	67.68	5.60

N*: Number of the specimens

C GIC: Conventional GIC

RM GIC: Resin-modified GIC

H CR: Hybrid composite resin

M CR: Microfilled composite resin

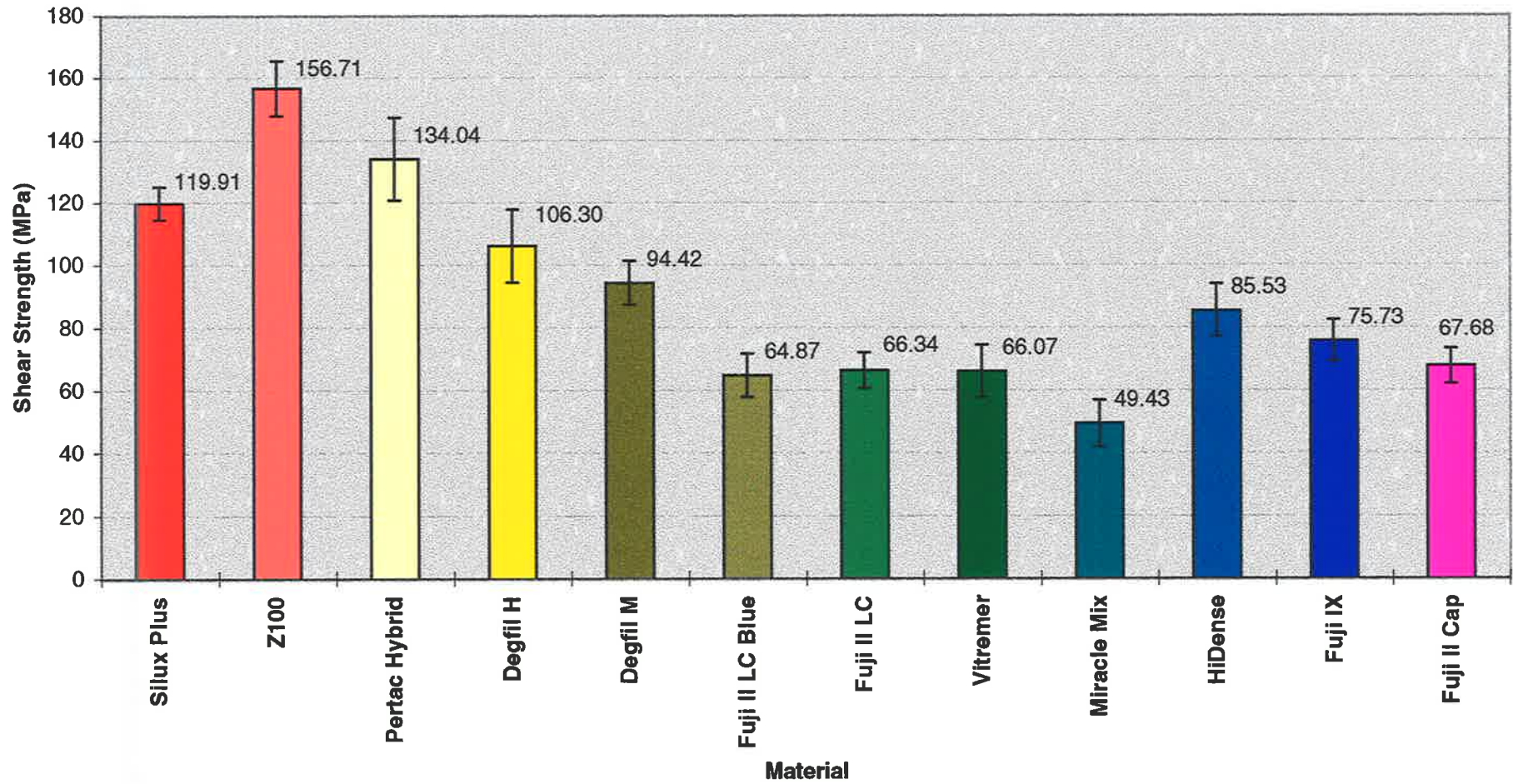


Figure 6.1

Comparison of various GICs and CRs

The mean shear strength of five CRs and seven GICs stored in water for one week.

* Error bars represent the standard deviation.

Table 6.3

The description of the materials, the sample size, the mean strengths and the standard deviations of the CR group and the GIC group.

Group	N*	Mean strength (MPa)	SD
CR group	48	121.31	23.61
GIC group	67	68.05	12.49

N*: Number of the specimens

Table 6.4

The results of the statistical analysis of the shear punch strengths between the GIC group and the CR group. (One way ANOVA test)

Source	df*	SS**	F ratio	P value
Between groups	1	79336.74	245.70	< 0.0001
Within groups	113	36487.44		
Total	114	115824.18		

df*: Degree of freedom

SS**: Sum of Square

Table 6.5

The results of the statistical analysis of the shear punch strengths of each material. (One way ANOVA test)

Source	df*	SS**	F-ratio	P value
Between groups	11	108849.47	146.13	< 0.0001
Within groups	103	6974.71		
Total	114	115824.18		

df*: Degree of freedom

SS**: Sum of Square

6.5 Discussion

Numerous reports regarding the strengths of CRs and GICs have been published in the literature. The studies which reported data for the same materials used in this study are reviewed and listed in Table 6.6. These data are difficult to compare as the preparation methods of specimens and testing modes varied in each experiment. Especially when the testing modes are different, the conclusions drawn may be quite different. For example, Mitra and Kedrowski (1994) studied the strength of GICs for both diametral tensile and compressive strength. In their study, the diametral tensile strengths of conventional GICs (Fuji II Cap) were nearly one third of those of the RM GICs (Vitremer and Fuji II LC) but the compressive strength of Fuji II Cap was only 10-20% lower than that of the RM GICs. Another example is the study reported by Hammesfar (1994). In his study, Fuji II LC showed nearly half the flexural strength while the results from other tests showed less discrepancy between the two materials. These kinds of differences may sometimes mislead clinicians as the studies often adopt one single physical test to draw a conclusion about the superiority of materials.

In the case of this study, the data should be carefully interpreted and perhaps the best way to assess the materials based on a single physical test may be not evaluating individual values, but "ranking" the materials. The reviewed results from the previous literature also support this view because the ranking of materials did not change when the testing modes differed (McCrary and Powers, 1992, 1993; Cattani-Lorente *et al.*, 1993; Hammesfar, 1994; Mitra and Kedrowski, 1994; Suzuki *et al.*, 1995).

The ranking of the materials considered in this study was interesting as within the GIC group, the RM GICs were expected to show better strengths due to the inclusion of resin. Instead, HiDense showed the strongest value of all the GIC materials. In the initial stage, the strengths of RM GICs may have shown higher values compared to the conventional GICs due to the quick set, but the tested RM GICs (stored in water for one week) did not particularly show consistently higher strengths than the conventional GICs.

Table 6.6 The reported strengths of the same materials tested in this study using various testing methods

Authors	Materials	Strength (MPa)	Mode of testing
Willems <i>et al.</i> (1992)	Z-100	448	Compressive
	PertacHybrid	450	
McCrary and Powers (1992,1993)	Fuji II LC	19.3	Diametral tensile
	Vitremer	40.8	
	Fuji II LC	151	Compressive
	Vitremer	198	
Cattani-Lorente <i>et al.</i> , (1993)	Fuji I	119.8	Compressive (24 hours)
	Fuji II	158.9	
	Fuji I	5.5	Tensile (24 hours)
	Fuji II	8.7	
	Fuji I	2.7	Flexural (24 hours)
	Fuji II	3.5	
Hammesfar (1994)	Fuji II LC	170	Compressive (24 hours)
	Vitremer	203	
	Fuji II LC	35	Diametral tensile (24 hours)
	Vitremer	40	
	Fuji II LC	38	Flexural (24hours)
	Vitremer	76	
Mitra and Kedrowski (1994)	Fuji II Cap	201 (15)	Compressive (1 week)
	Fuji II LC	214 (2)	
	Vitremer	256 (4)	
	Miracle Mix	150 (12)	
	Fuji II Cap	15.0 (2.5)	Diametral tensile (1 week)
	Fuji II LC	41.2 (1.4)	
	Vitremer	44.8 (1.5)	
	Miracle Mix	8.9 (1.3)	
Suzuki <i>et al.</i> (1995)	Fuji II	202	Compressive (24 hours)
	Fuji IX	220	
	Fuji II	209	Compressive (1 week)
	Fuji IX	241	
	Fuji II	16	Diametral tensile (24 hours)
	Fuji IX	22	
Uno <i>et al.</i> (1996)	Fuji II LC	15.1 (1.7)	Diametral tensile (1 week)
	Vitremer	15.7 (2.8)	

6.6 Conclusion

The information obtained from this part of the study was useful for comparing the shear punch strengths of GICs and CRs available in Australia. The shear punch method appeared to be advantageous in allowing the comparison of the physical strength of both CR and GIC restorative materials. All CRs showed a higher strength than GICs although in each group the strengths showed a wide variation. Interestingly the RM GICs did not show significantly greater strength than the conventional GICs and therefore the search for stronger GICs should continue in future.

Part II - Comparison of Three GIC Restorative Materials

Chapter 7 Preliminary Studies to Assess GIC Wear Resistance

7.1. Overview

In the previous chapters, the characteristics of the various GICs are discussed and compared with CRs. In the following two sections (Chapter 7 and Chapter 8), three different types of commercially available GICs (Fuji II Cap, Fuji II LC and Fuji IX) were considered and wear against enamel was assessed using a wear machine designed in the University of Adelaide. As there was no universal standard testing to evaluate *in vitro* GIC wear resistance, a series of preliminary studies were carried out to establish a method to assess *in vitro* GIC wear. The following preliminary studies are described in this chapter:

Preliminary Wear Study One to Three: Specimen designs

Preliminary Wear Study Four: Effect of enamel surface

Preliminary Wear Study Five and Six: Effect of water evaporation

7.2. Introduction

In this study, the wear simulation machine designed in the University of Adelaide was used to assess GIC wear characteristics. Previously, both enamel and dentine wear using this machine were studied by Kaidonis *et al.* (1995) and Partington *et al.* (1995) and the experimental procedures have been well established. For this study, some modifications were necessary to study wear in dental materials using this machine. Firstly, the brittle nature of materials such as GICs required special considerations. Several specimen designs were investigated. Secondly, information relating to changes in the enamel specimens during the experiment were considered using SEM and the way to control experimental variables was also investigated. Thirdly, the influence of the water content on the specimen was studied. As the wear was measured by weight, assessment of the weight change due to the water evaporation from the

specimens over time was mandatory.

Based on the information studied in these preliminary studies, the protocol for the final experiment was established. The details are discussed in Chapter 8.

7.3 Materials and methods

Materials

The materials and their manufacturers details used in the experiments of Chapter 7 and 8 are described in Table 7.1. It was known that the average power particle size for all these products was 4 micrometers (GC personal communication, 1995). Fuji II LC was a RM GIC which contains HEMA in its liquid. Fuji II Cap was a Type II restorative GIC and the capsulated version of Fuji II. Fuji IX was described as "reinforced GIC" by the manufacturer and was especially developed for the A.R.T. technique. More details regarding the A.R.T. technique are described in Chapter 8.

Table 7.1

Manufacturers details and types for the three GICs used in Chapter 7 and 8

Material	Manufacturer	Batch #	Description	P/L
Fuji II Cap	GC Co., Tokyo Japan	300391	C GIC/capsulated	2.7g/1.0g
Fuji II LC	GC Co., Tokyo Japan	041045	RM GIC/capsulated	3.0g/1.0g
Fuji IX	GC Co., Tokyo Japan	110341	C GIC/capsulated	3.6g/1.0g

C GIC: Conventional GIC

RM GIC: Resin-modified GIC

Wear machine and its operation

This study was conducted using the tooth wear machine designed and built in the University of Adelaide. The machine is shown in Figure 7.1. The apparatus consisted of a stainless steel fixed lower section and a movable upper section. Each section had a removable specimen holder. Specimens were attached to these holders by tightening a laterally directed grub screw which created an indentation on the specimen mounting. Accurate repositioning of the specimens were ensured every time when specimens were released from the holder by the alignment of this indentation. The GIC material was placed in the lower holder of the wear machine and an enamel specimen was placed in the upper holder (Figure 7.2). The enamel specimen was rubbed against the fixed GIC specimen with a uni-directional movement for a specific duration.

The standard speed during the machine operation was 80 cycles per minute simulating normal chewing cycles. An analog counter fixed to the resultant drive of the gearbox recorded the number of cycles of the machine. The inherent weight of the upper movable member was 3.2 kg. The machine was designed such that the load could be adjusted between 0.25kg to 16.2kg. Continuous lubrication was applied between the rubbing surfaces using a hospital intravenous drip system. A 0.6 mm thick aluminum cement spatula was inserted between the enamel and GIC specimens at the closest position in setting up the GIC specimen to standardize the distance between the enamel and GIC specimens. Before and after specified numbers of cycles of the experiment, specimens were removed from the wear machine and dried carefully with a paper towel, and maintained at room temperature for a prescribed time before measuring the weight. An electric scale (ER-182A Electronic Analytical Balance, AD Company limited, Tokyo, Japan) was calibrated according to specifications and tested using known weights prior to the weighing of the specimen. The balance was kept on a steady stand away from vibrations, and the specimens were weighed in a closed chamber to avoid the effect of air currents. More detailed description of the apparatus is given in Kaidonis (1995).

Enamel specimen construction

A previous study by Kaidonis (1995) established the manner of constructing enamel specimens and creating enamel facets. Extracted teeth were cleaned and longitudinally sectioned mesio-distally. The pulpal tissue was removed and the specimens stored in water. A 19 mm diameter and 18 mm length of plastic cylinder was cut and smoothed and an SEM stud was cemented on one end with Araldite™ epoxy resin (Ciba Geigy). The adhesive was left to harden over night. Excess resin, and rough edges were removed. Sectioned teeth were cemented to the SEM stud and both the adhesive and the tooth surface were covered with nail varnish. Enamel specimens were then placed on both upper and lower members of the wear machine. Enamel facets were created with the machine operating 80 cycles per minute for one hour with a load of 9.95 kg under water lubrication. The enamel specimens prepared in this way are subsequently described as the 'enamel specimen' in each section .

GIC specimen construction

As no previous studies of "material" wear had been conducted with this wear machine, three preliminary specimen designs were considered to establish an appropriate model for the final experiments. They are discussed in the following section.

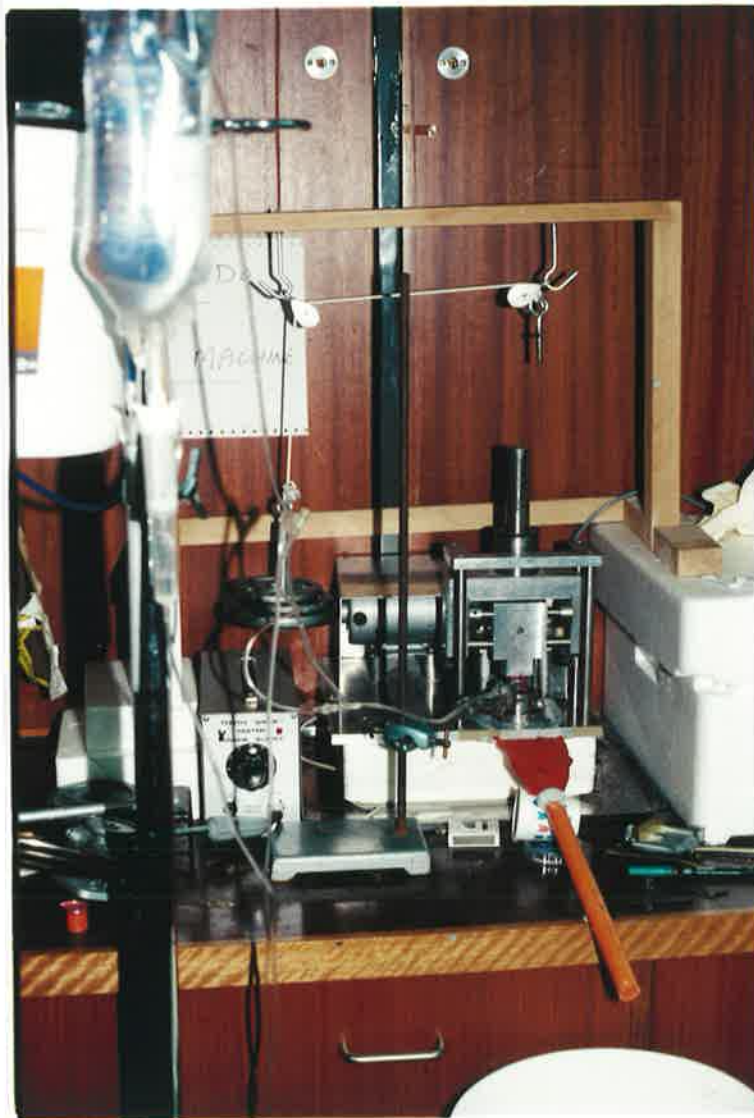


Figure 7.1

The view of the wear machine designed in the University of Adelaide. The apparatus consisted of the stainless steel fixed lower section and a movable upper section.



Figure 7.2

Specimens were attached to these holders by tightening a laterally directed grub screw which created an indentation on the specimen.

7.4 Preliminary wear studies: the GIC specimen design

7.4.1 Preliminary Wear Study One-Design A

7.4.1.1 Introduction

Two Fuji II LC specimens were made for a preliminary study as a modification of the enamel specimen design described by Kaidonis (1995).

7.4.1.2 Materials and methods

To create two GIC specimens, a 6 mm diameter and 2 mm deep indentation was drilled in the center of each SEM stud and four indentations with an inverted cone bur were added for retention. Plastic rings with approximately 7 mm diameter and 2 mm height were prepared to form 4 mm thick GIC specimens. The 2 mm thick first layer of Fuji II LC was placed in the hole and cured for 40 seconds. The process was repeated in the secondary increment of the material to create an approximately 4 mm thick specimen. In curing of this final increment of GIC, a mylar strip was placed on the top to create a flat surface. After removing a plastic ring around GIC, both the cement and a SEM stud except the flat surface were immediately coated with nail varnish to limit water uptake. The mylar strip was then removed and the specimens were kept in water for one week. One enamel specimen was made in the manner described in Section 7.3.

After storage, the first GIC specimen and the enamel specimen were carefully dried with a paper towel, and left for 15 minutes at room temperature and the weight of each specimen before the wear experiment was recorded. After exactly 30 minutes of wear under water with a total load of 6.4 kg, both the GIC and enamel specimens were removed from the machine, and dried in the same manner and the weight loss was measured. During this operation, the dial of the wear machine was set to 80 cycles per minute, however the actual wear cycles were recorded individually from the counter in this study. For the second GIC specimen, the same procedure was repeated using the same enamel specimen but the applied total load was 12.5 kg instead of 6.4 kg.

7.4.1.3 Results

A summary of the results is described in Table 7.2. The weight change of the GIC specimens and the enamel specimen before and after the wear experiment suggested that the GIC specimen showed more wear than the enamel specimen.

Table 7.2

The weight loss of the two Fuji II LC and one enamel specimens worn for 30 minutes at 80 cycles per minute under two different loads.

Load (kg)	Cycles	GIC weight loss (mg)	Enamel weight loss (mg)
6.4	2814	0.28	-0.0003
12.5	2764	0.23	0.0002

7.4.1.4 Discussion

This preliminary study supplied useful information to identify the problems related to the GIC wear study. Firstly, the method to control the moisture should be further studied. In the wear study by Kaidonis (1995), enamel specimens were dried for 2 hours before weighing. By that stage the weight was stable. However, it was not adequate to adopt this method for GIC as the specimens showed initial cracks even after 13 minutes. Further details regarding this are discussed in Section 7.6.

Secondly, after one week, extensive corrosion of the SEM studs was recognized beneath the nail varnish. Some parts of the nail varnish were lifted from the aluminum surface due to the oxidised substance. This was found in both enamel and GIC specimens, but the GIC specimens showed more corrosion than the enamel specimen perhaps because of the moisture from GICs. This was not a desirable experimental condition as the weight would have been influenced by the moisture trapped between nail varnish and the corroded surfaces.

Thirdly, the effects of an applied load on the specimens was unknown. The results of this study suggested that a doubled load did not appear to affect the weight loss. The reason for this was not clear but the water evaporation from the specimens might have contributed to a larger part of the weight loss than that from the actual wear. Perhaps for the same reason, one of the enamel specimens showed a small increase in weight.

This experiment also revealed that it was inappropriate to rely on time as the best method to

control the number of cycles. Although the expected total number of cycles from 30 minutes of operation was 2400 cycles, both experiments showed approximately 10% more cycles. Therefore, for the rest of the experiments, a specified number of cycles recorded by a counter was used instead of the duration of the wear operation with a controlling dial set with 80 cycles per minute.

7.4.1.5 Conclusion

This first protocol was not suitable to measure wear rate of GIC although this experiment supplied important information for the desired GIC specimen design.

7.4.2 Preliminary Wear Study Two-Design B

7.4.2.1 Introduction

To overcome the identified problems such as the corrosion problems of the mounting material, a second specimen design using a stainless washer was developed.

7.4.2.2 Materials and methods

The following mounting apparatus was assembled for each specimen. A plastic cylinder described in the previous section was prepared and a stainless steel washer was cemented to one end and dried. A piece of an adhesive tape was placed to seal the bottom of the steel washer hole, and GIC was placed into this hole. Three groups (eight specimens of each) of Fuji II Cap, Fuji IX and Fuji II LC were made. On the top of the specimen, a mylar strip was placed and a glass slab was pressed to create a flat surface. At the same time, GIC was pressed out on the bottom after this procedure, and this part provided retention of the material. A specimen approximately 3 mm thick was made by this procedure. Nail varnish was painted all over except the top flat surface to limit water absorption (Figure 7.3). Specimens were stored in water for one week.

Three enamel specimens were made in the manner described previously, and each enamel specimen was rubbed against each group of the GIC specimens. The machine was operated at a load of 12.5 kg for 80 cycles per minute for 1000 cycles under water lubrication.

The GIC specimens which survived without fracture for 1000 cycles were recorded as 'survived specimen', and fractured specimens were recorded as 'cracked specimen' and were removed from the wear machine immediately after failure was detected. The percentages of survived specimens were calculated.

7.4.2.3 Results

The results of this study are described below (Table 7.3). All Fuji II LC specimens survived under this stress and no crack propagation was observed on the surface of the survived samples. Fuji II Cap showed the highest frequency of shattering in the early stage of loading. In this group, the fractures extended through whole the specimen, and catastrophic failure was observed.

Table 7.3

The number (no.) of the 'cracked' and 'survived' specimens of the three GICs in Preliminary Wear Study Two (wear at 80 cycles per minute with the load of 12.5kg)

Materials	Total no. of samples	No. of 'cracked' sample	No. of 'survived' samples	Percentage of 'survived' samples
Fuji II Cap	8	4	4	50%
Fuji IX	8	2	6	25%
Fuji II LC	8	0	8	100%

Figure 7.3

The view of the Specimen Design B. A stainless steel washer assembled with a plastic cylinder was used for the mounting GIC. Nail varnish was painted all over except the top flat surface to limit water absorption.



7.4.2.4 Discussion

It was interesting to see each material showed a different tolerance to the same load. According to this result, Fuji II LC appeared to be the strongest of the three materials. However, this result should be carefully interpreted as several factors appeared to influence this observation. One factor might have been related to voids in the specimens. Of the three materials, only Fuji IX was hand-mixed while the other two were capsulated versions. Mount (1994b) stated that hand-mixed versions bear a greater variation in the size of porosity, while capsulated versions created uniformly smaller voids. The higher failure found in Fuji IX compared to Fuji II LC might have been caused by bigger voids in Fuji IX which caused stress concentration and started crack formation.

The other factor might have been the quality or/and morphology of the enamel specimens. When the rubbing surface of the enamel specimen is smaller, the force per unit area is greater and thus there would be more chance for the fracture. It was therefore difficult to be confident in the ranking of the materials from this preliminary study as the enamel specimens used in each material were different. A study to control enamel specimens was necessary for the final experiment. It is discussed in Section 7.5.

7.4.2.5 Conclusion

Although the corrosion problem seen in the first preliminary design had been improved, the specimen design of this study was not adequate to assess the GIC wear as many samples in this design did not withstand the stress during the wear operation. To avoid the catastrophic failure, a design supplying additional support of the materials was desirable.

More improvement of the design was necessary to be successful to assess the wear rate of the materials.

7.4.3 Preliminary Wear Study Three-Design C

7.4.3.1 Introduction

Based on the information which was gained from the first two preliminary studies, usage of a polyethylene material which had a property of 0% water uptake was considered. Since this was a preliminary study, only the two materials which showed catastrophic failure in Preliminary Wear Study Two were selected.

7.4.3.2 Materials and methods

A rod made of high molecular weight polyethylene (Tivar 1000™) was turned on a lathe to produce the 22 mm high by 19 mm diameter specimen holders. This material was reported as

having 0% water up-take by the manufacturer. A cavity was prepared in the centre of one end with a 8.5 mm diameter drill piece, approximately 3.5 mm deep. Additional mechanical retention was achieved by creating undercuts with an inverted cone diamond bur. In this design, GIC materials were supported by Tivar 1000 everywhere except the rubbing surfaces.

Two samples each of Fuji II Cap and Fuji IX were mixed according to the manufacturer's instructions and syringed into the cavities. A mylar strip was placed on the top of the mixed GIC, and pushed to the level of the Tivar surface with a glass slab to create a flat surface (Figure 7.4). After an initial set, the samples were kept in a humidor at 100% relative humidity at room temperature for one week. Excess material was trimmed before measuring the original weight. The GIC specimens were rubbed against a single enamel specimen in the following sequence: two Fuji IX specimens and then two Fuji II Cap specimens (in sequence of Specimen A,B,C,D). A 9.95kg load at 80 cycles per minute was applied during the machine operation. The weight was measured before and after every 1000 cycles in the manner described, with the specimens dried for 3 minutes at each stage in this experiment. The weight change was monitored for 10000 cycles.

7.4.3.3 Results

The accumulated weight changes of each GIC specimen are described in Figure 7.5. One Fuji IX specimen showed a small chipped edge after the first 1000 cycles, otherwise, there were no cracks or fractures noted.



Figure 7.4

The view of the specimens (Design C) using Tivar 1000™.

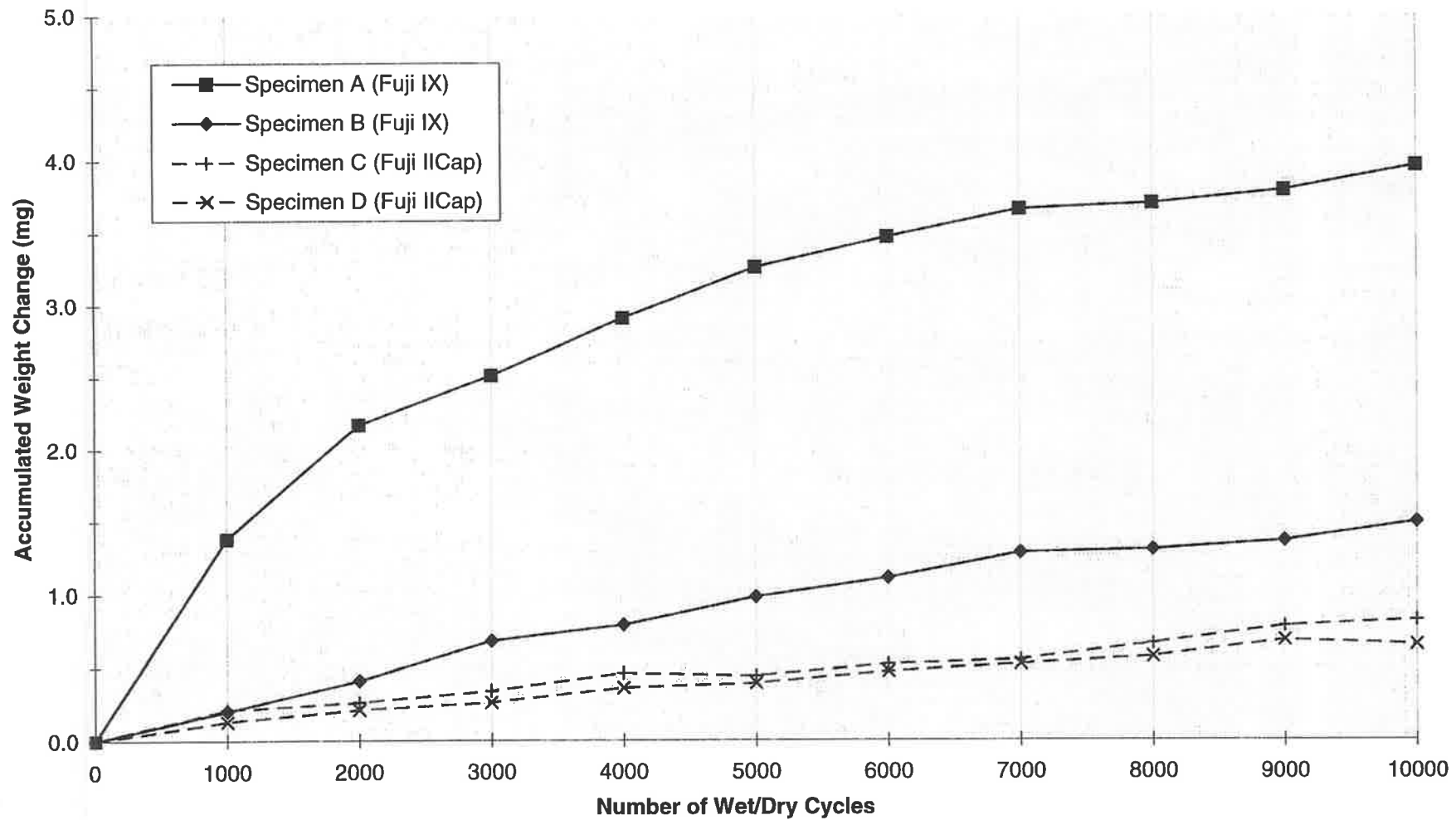


Figure 7.5
Accumulated Weight Changes
 The accumulated weight changes of each GIC Specimen up to 10000 cycles



7.4.3.4 Discussion

This design provided satisfactory results. The size of the drilled hole was so uniform that it was possible to control the volume of GICs. This meant that the moisture content within each GIC specimen and the surface exposed to air from which the moisture would evaporate were also controlled. The chance of fracture was minimized as the material was well supported against lateral forces. GIC specimens were kept in a humidior at room temperature in this experiment. While Fuji IX specimens did not show any significant visual change before and after the storage, Fuji II Cap specimens showed a whitish color change which was considered to be an indication of the hydration of the cement. This observation matched the results discussed in Section 4.6 (Preliminary Study Three-Storage conditions and the shear strength). Based on this information, it was decided to store the samples in water to avoid dehydration of GICs in the final study.

In this experiment, the same enamel was continuously worn against all four GIC specimens. One important finding was that the longer the enamel specimen had been rubbed against GICs, the smoother and wider the enamel surface became and thus the lower the wear rate was. The protocol needed modification to control the enamel specimens and more experiments relating to control of the enamel specimen are discussed in the following section.

7.4.3.5 Conclusion

The third design succeeded in creating standardized GIC specimens for wear assessment. All the GIC specimens for the rest of the wear studies were made in the manner explained in this section and stored in water for one week unless otherwise stated. The specimens prepared in this way are subsequently described as the 'GIC wear specimen' in each section .

7.5 Preliminary Wear Study Four: Effect of enamel surface

7.5.1 Introduction

The results of the earlier preliminary wear study suggested that the GIC wear rate decreased when the same enamel specimen was continuously used. In this section, details of the enamel surfaces and the wear of GIC specimens were investigated by SEM using a replica technique. Based on this information, the method for standardizing the enamel specimens are discussed.

7.5.2 Materials and methods

Seven Fuji II LC specimens stored in water for one week and two enamel specimens were used. They were constructed in the manner described. The GIC specimens (in sequence of Specimen G1, G2, G3, G4, G5, G6, G7) were worn for 7000 cycles against a single enamel specimen

(Specimen E1) and the weight change was monitored in the manner described. The applied load was 9.95 kg at a speed of 80 cycles per minute with water lubrication.

At several stages the Specimen E1 surface was recorded using a replica technique. Polyvinyl siloxane impression material (Extrude, Kerr Manufacturing Company Romulus, USA) was used to obtain impressions of the surface of the enamel specimen. Epoxy resin dies (Ivoclar Epoxy Die Material, Ivoclar, Schaan, Liechtenstein) were poured into the impression. Epoxy dies were mounted and coated for the SEM (Philips XL 20, Philips, Holland) analysis.

The weight change of each of the GIC specimens was recorded every 1000 cycles after being carefully wiped with a paper towel and dried at room temperature for 3 minutes. The enamel specimen (Specimen E1) was continuously worn against four GIC specimens (in sequence of Specimen G1, G2, G3, G4) so that Specimen E1 underwent a total of 28000 cycles.

After this procedure, the surface of Specimen E1 was then manually roughened against 100 grit silicone carbide paper by rubbing 10 times. This process (enamel wear) is subsequently described as "scratching" in this study. Another GIC specimen (Specimen G5) was worn against this "scratched" Specimen E1.

After this procedure, the enamel specimen was worn against another flat enamel specimen (Specimen E2) using the wear machine (for 15 minutes with the load of 9.95kg in a speed of 80 cycles per minute). This process (enamel wear) is subsequently described as "roughening" in this study. After wearing Specimen G6 against the "roughened" Specimen E1, Specimen E1 was again "roughened" and Specimen G7 was then worn against it.

7.5.3 Results

The results of the GIC wear are shown in Figure 7.6. The graph shows that the amount of the wear decreased gradually from Specimen G1 to G4, but that the GIC wear rate increased after the enamel surface was "scratched" with sand paper. The SEM views of the following four stages are shown in Figure 7.7: the original enamel specimen surface before wearing against Specimen G1 (View A), the highly polished enamel surface after wearing against Specimen G4 (View B), the "scratched" enamel surface before wearing against Specimen G5 (View C), the "roughened" enamel surface before wearing against Specimen G6 (View D). An extremely smooth enamel surface was observed in View B, accompanied with the decreased wear rate. Sudden increase of the wear rate was observed after the enamel surface was "scratched". View D showed the similar view to View A.

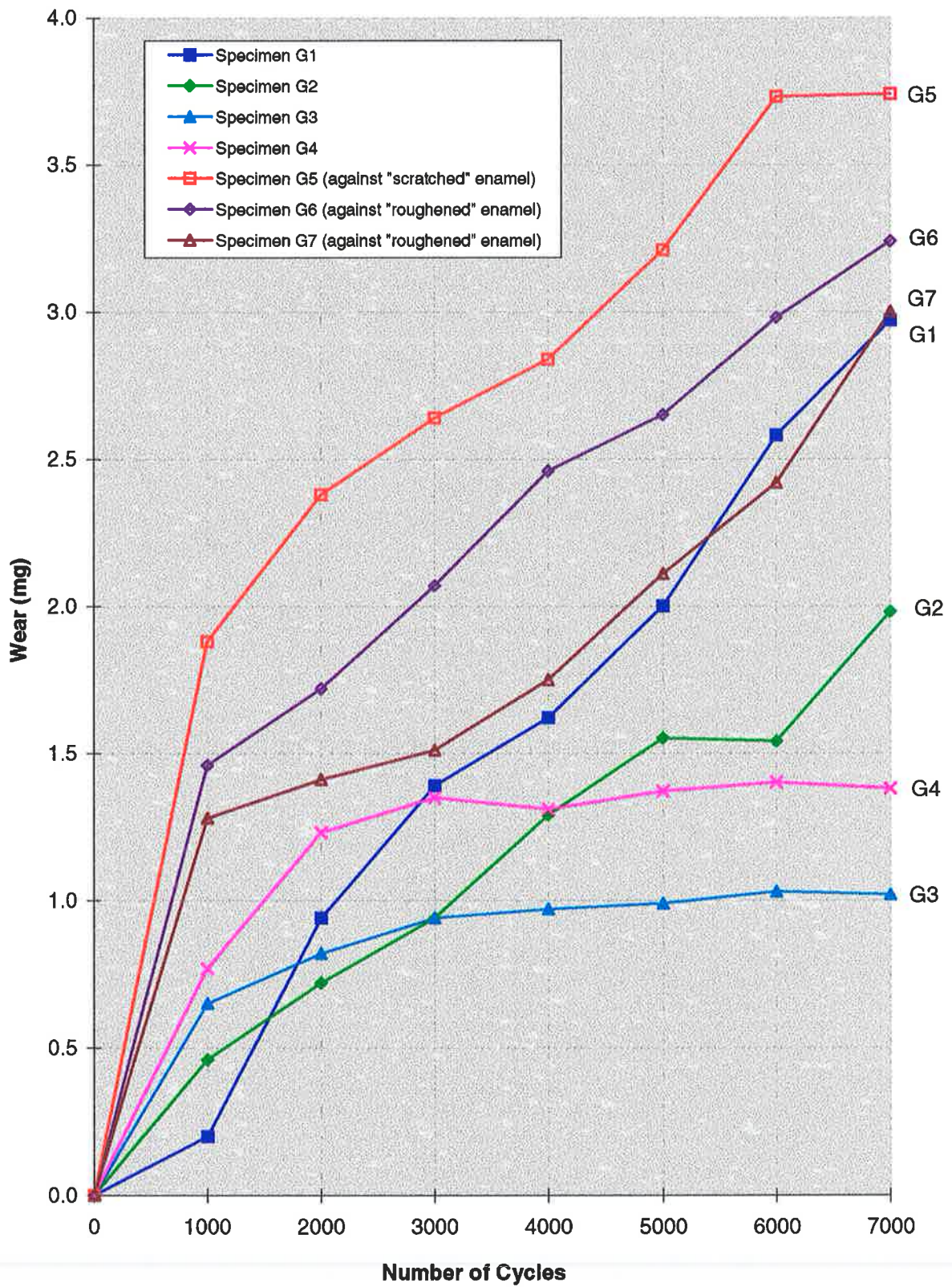
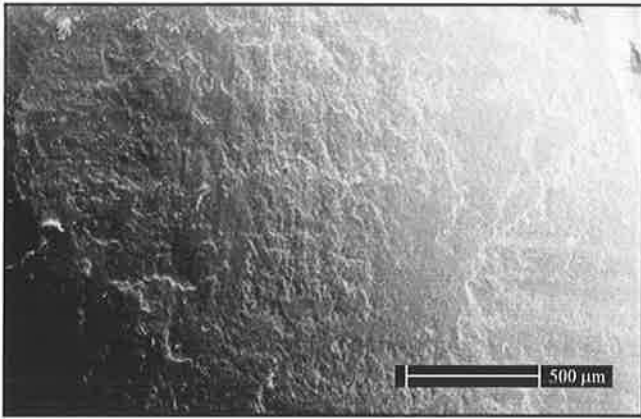
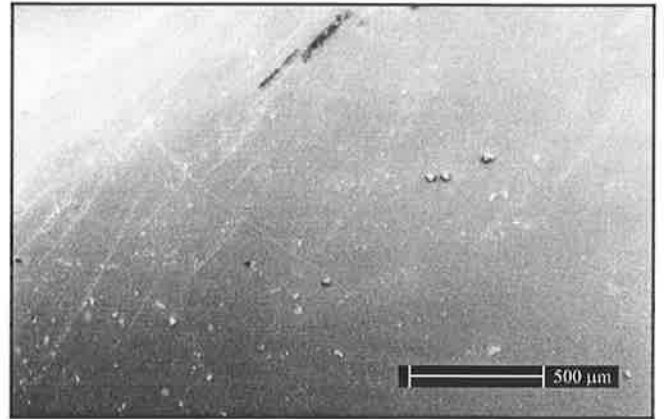


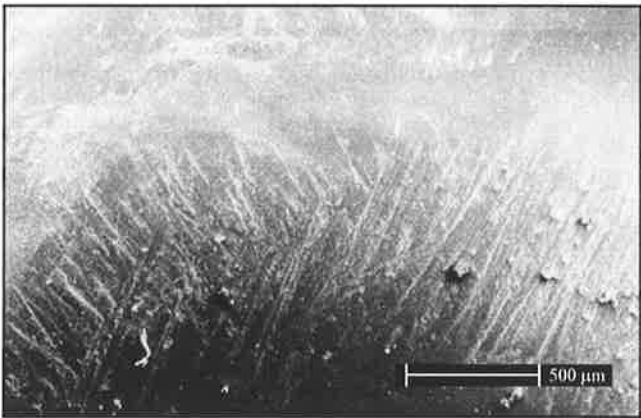
Figure 7.6
Wear of Fuji IILC with different surface treatments.



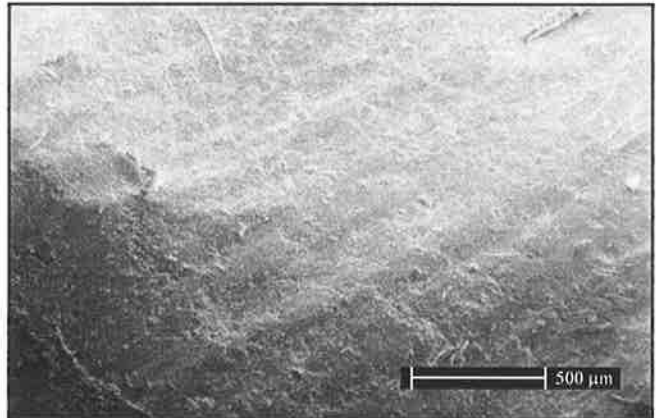
View A
The original enamel specimen surface before wearing against Specimen G1.



View B
The highly polished enamel surface before wearing against Specimen G4.



View C
The "scratched" enamel surface before wearing against Specimen G5.



View D
The "roughened" enamel surface before wearing against Specimen G6.

Figure 7.7 SEM Views.

The SEM views of the enamel specimens at the four stages of Preliminary Wear Study Four.

7.5.4 Discussion

Many wear studies adopt the concept of two stages of the wear process when assessing the wear rate of materials. The wear rate is not found to be constant in the early stages and as a result, the data obtained after a certain time from the onset of wear ("steady-state" wear) are usually used to determine the wear characteristics of materials (McKinney *et al.*, 1988). Specimen G3 had shown almost no wear against the enamel specimen towards the end of the experiment, but when a different specimen (Specimen G4) started to wear, the material showed a greater wear rate over the first 2000 cycles then again showed no sign of further wear after that period. Based on this information, the wear data for the first 2000 cycles were eliminated to obtain the wear rates at a "steady-state" of wear, and the protocol of using only the data between 2000 cycles to 10000 cycles to determine the wear rate was adopted in the final study.

The results of this study suggested that the surface condition of enamel significantly influenced the wear rate of GICs. The enamel specimen which experienced repeated wear against GIC showed a highly smoothed surface, and at the same time the GIC wear rate decreased. It was obvious from the SEM views that the texture of this highly smooth surface was different to the original surface. To assess wear characteristics in each material, it was important to standardize the enamel specimen condition. Although it was easy to create a standardized rough surface using sandpaper, this kind of surface was not likely to be seen in the clinical situation. The "roughened" surface rubbed against a flattened enamel appeared to be reproducible and more clinically relevant, thus this method was adopted in the final protocol. Another problem in standardizing the enamel specimen was the variation in contact area with the enamel surface. Naturally the contacting area of the enamel specimen increased in size as the number of cycles increased.

Based on the various information obtained from this preliminary study, the following method was adopted as the final protocol:

Two morphologically similar third molars from the same patient were carefully selected. Each tooth showed a symmetric outline so that after each tooth was split, four morphologically similar samples were obtained. They were mounted by the method described previously and were rubbed against the flat enamel for 15 minutes using the wear machine. By this method, homogenous enamel specimens with similar geometry and quality were created. Each enamel specimen was used to assess each six specimens of the same material. An enamel surface was 'roughened' by another flattened enamel surface to standardize the surface texture each time a GIC specimen was changed.

7.5.5 Conclusion

The study showed that different enamel surface texture influenced the GIC wear rate. To standardize enamel surface texture, "roughening" an enamel surface by rubbing against another flat enamel surface for 15 minutes using the wear machine was considered to be the best way.

7.6. Preliminary wear studies: Effect of water evaporation

7.6.1 Preliminary Wear Study Five

7.6.1.1 Introduction

This experiment was conducted to determine the time each specimen must stand in air before weighing. Since GIC specimens contain a significant amount of water, the issue of water evaporation and time elapsed before weighing was an important consideration in the design of the final experiment. Two preliminary studies regarding water evaporation were conducted to control experimental variables in the final wear assessment.

7.6.1.2 Materials and methods

GIC specimens were made using Tivar 1000 in the manner described in Section 7.4.3. Two samples each of three materials stored in water for one week were used in this study. Specimens were removed from water and carefully dried with a paper towel and placed on the electronic scale table. Weight change was recorded every minute for 20 minutes (Procedure A). After this procedure, specimens were soaked in water for 40 minutes, and then again dried with a paper towel carefully and the weight change was monitored for 10 minutes (Procedure B). The specimen were then soaked in water for another 40 minutes and the weight change was again monitored for 10 minutes (Procedure C).

7.6.1.3 Results

The results of this study were shown in Fig 7.8, Fig 7.9 and Fig 7.10. All the specimens showed continuous weight loss over time.

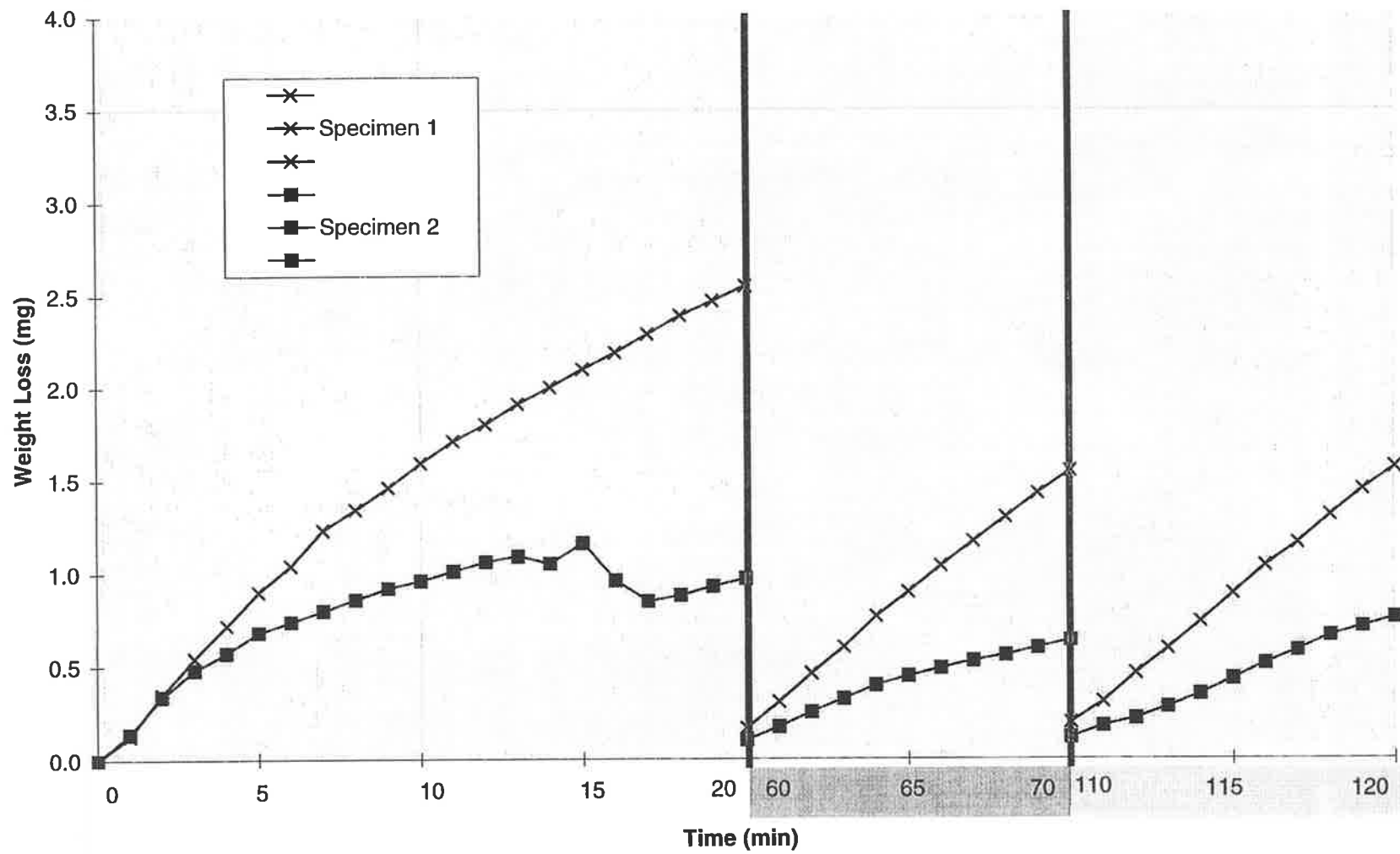


Figure 7.8
 Preliminary Wear Study Five *Water Evaporation (Weight Loss) of Fuji II Cap*

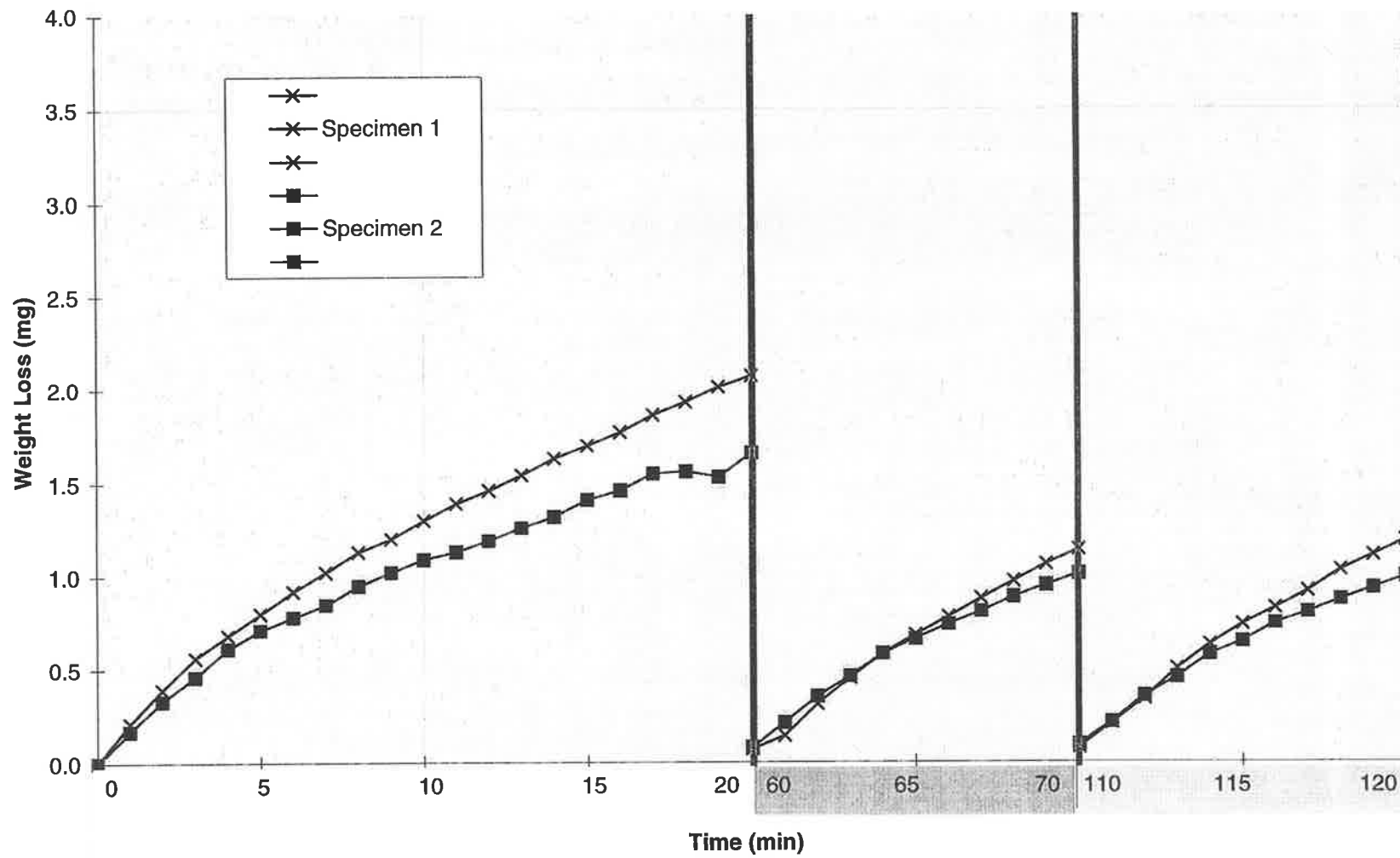


Figure 7.9
 Preliminary Wear Study Five *Water Evaporation (Weight Loss) of Fuji II LC*

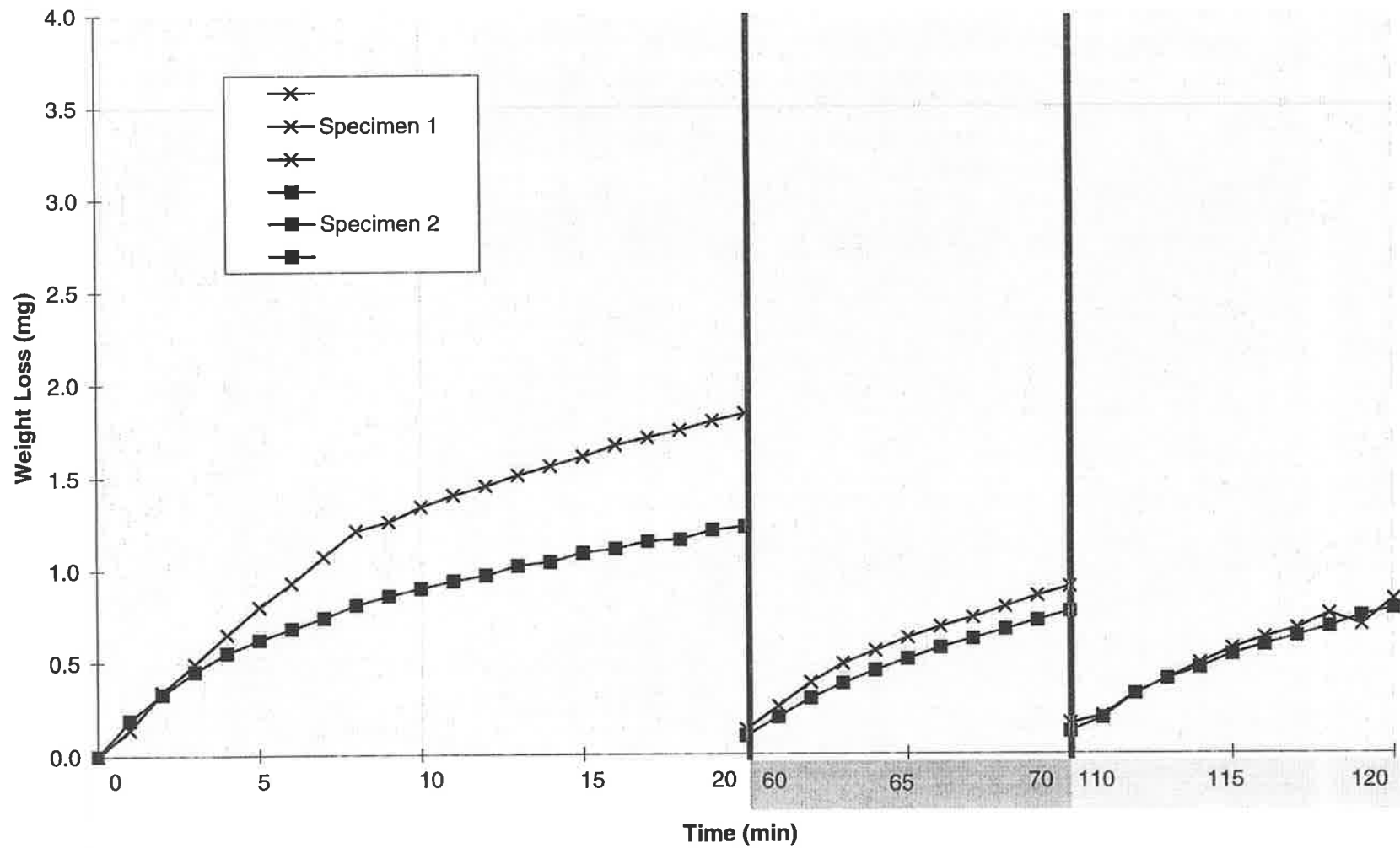


Figure 7.10
 Preliminary Wear Study Five *Water Evaporation (Weight Loss) of Fuji IX*

7.6.1.4 Discussion

In general, for experiments involving the weight change of 'wet' specimens, it would be best to leave materials as long as possible to establish a stable value to avoid the errors arising from different amounts of moisture on the surface of the specimen. At least it was considered that the longer the specimens stood, the more consistent the results became because the weight change was slowing down over time. However, in case of GICs this was inappropriate as the material showed obvious desiccation after a relatively short time and cracks appeared.

According to the results, none of the specimens reached a stable weight even after 20 minutes and continuous weight loss was observed although the rate of change reduced over time. After the specimens were dried in Procedure A, none regained their original weight even after they were soaked in water for 40 minutes. However, the weights at the beginning of Procedure B and C for the each sample were similar value. The two curves for these stage were also very similar. The reason for this behaviour was not entirely clear, but it could be explained by two stages of the desiccation process in GICs. Dehydration for 10 minutes might be 'recoverable' by immersing in water because the water lost in this period could be mainly moisture from the surface. Once GICs experienced excessive dehydration, the weight loss might not be recoverable as it might involve water from the GIC itself.

Due to the standardized size of each specimen, and the fact that there is no water absorption in Tivar 1000, it was assumed that the amount of moisture from the surface of a mounting material was well controlled. However, some variation was seen in each material. Initiation of a surface color change was observed within 4 minutes in Fuji II Cap, 6 minutes in Fuji IX, and 13 minutes in Fuji II LC. Crack formation was apparent after 7 minutes in Fuji II Cap and 8 minutes in Fuji IX, but not seen in Fuji II LC specimens even after 20 minutes. Once one crack started, it tended to deepen and extend. Perhaps this crack resulted in an enlarged surface area exposed to the air, allowing further dehydration to proceed at an increasing rate.

In terms of crack formation, Fuji II LC appeared to be more stable. However, interestingly, the weight change in Fuji II LC were similar to that of Fuji IX. It was suggested that resin inclusion in GIC appeared to resist crack formation better than the other materials, but did not to stop water evaporation. Further research is necessary to obtain more information regarding this behaviour.

7.6.1.5 Conclusion

There were continuous weight changes in GIC specimens over 20 minutes when they were

exposed to air. In 4 minutes, one of the specimens showed the first sign of dehydration, and in 7 minutes, the first crack was observed. Based on this information, the standing time of 3 minutes before measuring the specimens was established as the protocol of the final study. This provided a degree of weight stability without significant deterioration of the material. This also allowed for the electronic scale to stabilize from the vibration created when the specimen was put on the scale table.

7.6.2 Preliminary Wear Study Six

7.6.2.1 Introduction

Based on the information from these previous studies, a protocol for the final study was established. The GIC specimens were removed from the machine after every 1000 cycle operation and dried with a paper towel and left to stand in air for 3 minutes. The machine was operated for 10000 cycles with water lubrication and thus GIC specimens would experience repeated wet and dry cycles in this process. The purpose of this study was to examine if weight change of the GIC specimens would occur after the repeated wet/dry cycles.

7.6.2.2 Materials and methods

Two samples each of three GICs stored in water for one week were used in this study. Specimens were removed from water and carefully wiped with a paper towel. After standing at room temperature for 3 minutes, the weight of the specimen was measured in the manner described, then immediately immersed in water for 10 minutes. This procedure was repeated 10 times for each sample and the weight change was monitored.

7.6.2.3 Results

The results of this study are shown in Fig 7.11. Although all specimens showed weight loss by the end of this procedure, the amount of loss varied between specimens. Fuji IX showed the most consistent results during this procedure and showed weight changes in the range of 0.2 mg over 10 wet/dry cycles. Fuji II LC showed slightly greater variation in one specimen, but the other specimen showed relatively stable value similar to Fuji IX. Fuji II Cap showed the most weight loss of the three materials (of the order of 1 mg over 10 wet/dry cycles). This result is discussed combined with the results of Chapter 8.

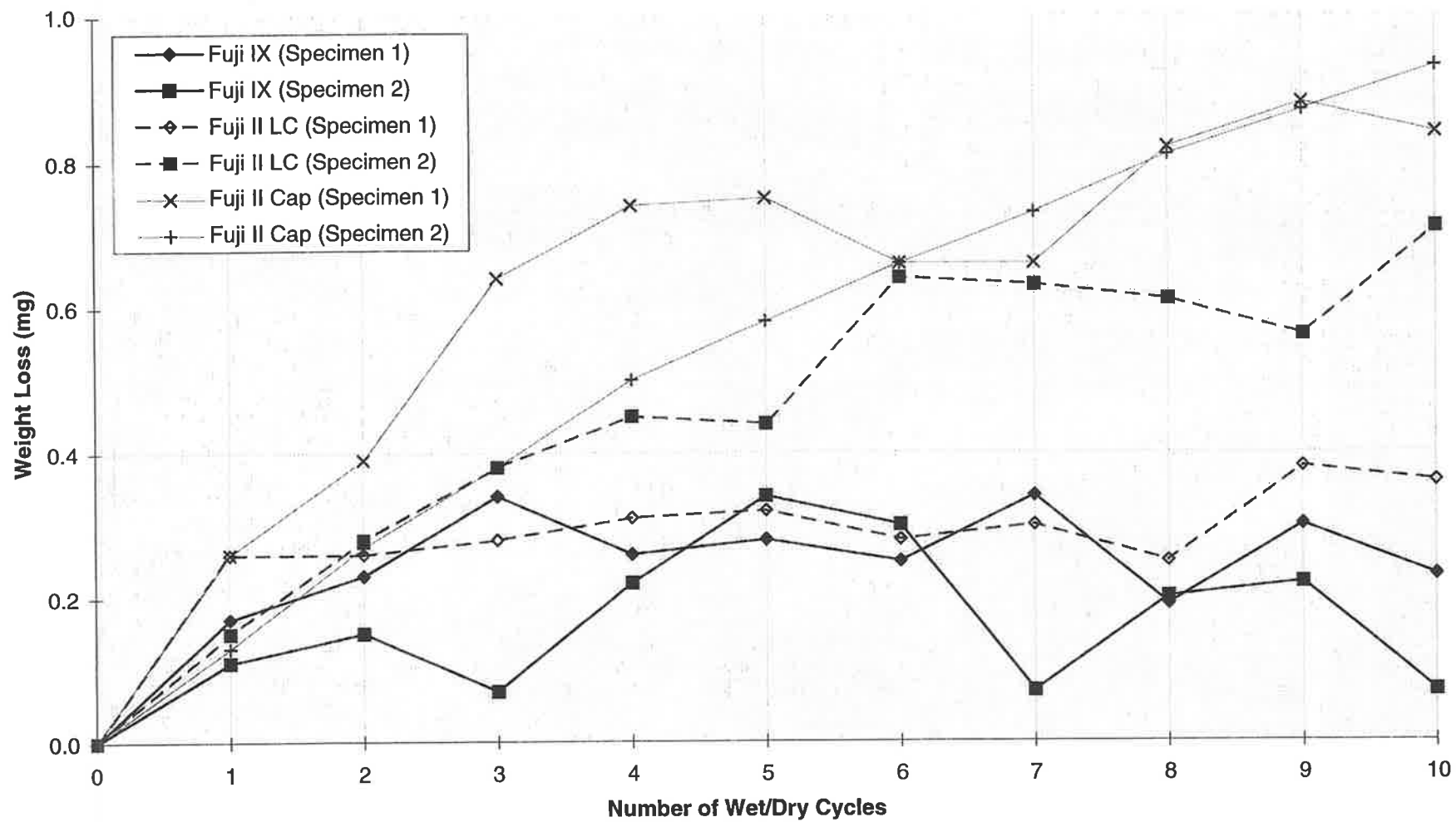


Figure 7.11
 Preliminary Wear Study Six Specimen Weight Loss over Wet/Dry Cycle repeated ten times.

7.6.2.4 Discussion

In this study, to draw some conclusion was difficult due to the limited sample size, however all specimens showed weight loss over 10 wet/dry cycles. This suggested that this wet/dry procedure caused slight dehydration of the cements. None of the specimens showed the evidence of color change or cracks after 10 wet/dry cycles. Fuji IX showed the smallest weight loss during the later stages of the procedure. Fuji II Cap showed the largest weight loss through this process although the rate of the weight change appeared to decrease over cycles. This suggested that Fuji IX appeared to show the highest resistance to the dehydration among three GICs. It should be noted that specimens stored in water for one week was used in this study and that specimens stored for longer may behave differently due to more conversion of loosely bound water into firmly bound water. This is discussed further in Chapter 8.

7.6.2.5 Conclusion

Of the three GICs, Fuji II Cap showed the greatest weight loss as a result of the 10 wet/dry cycles (approximately 1.0 mg) and all the specimens showed weight loss after these procedures with the Fuji IX specimens showing the most stable weight among three GICs.

7.7 Discussion

Often clinical wear studies are difficult, time-consuming, expensive, and involve complex factors such as variation of personal habits etc. (Harrison, 1976; Abell *et al.*, 1983). Therefore, most studies are conducted *in vitro*, and *in vivo* wear studies are less widely reported (Ekfeldt, 1989). Although a wide variety of sophisticated methods designed for simulated wear of dental materials have been attempted by several authors (see Section 3.6), none have been proven to be superior in simulating the clinical situation (McCabe, 1985; Sulong and Aziz, 1990). This is one reason why there is no standard test currently adopted by ISO. Many studies have reported material wear characteristics with specimens worn by contacting with various abrasives while few studies have reported wear against tooth structure. As the wear rate against enamel provides more clinically relevant information than data obtained from wear against abrasives, human enamel was used for assessing the wear characteristics of the GICs in this study. Preliminary studies were necessary to standardize the enamel specimens and the adopted final protocol appeared to achieve an appropriately controlled experimental condition.

Several designs were considered through the preliminary studies, and the final design gave a satisfactory reduction in experimental variation. Importantly, care to avoid dehydration should

be emphasized in handling not only conventional GICs, but also RM GICs. As previously discussed, the trend of the dehydration of the RM GICs was not particularly different from that of conventional GICs. Further studies are necessary to demonstrate this conclusively.

7.8 Conclusion

It is often difficult to establish a good correlation between findings *in vitro* and *in vivo* because it is very difficult to simulate the complex oral environment (McCabe and Smith, 1981). However the information obtained from this wear study using the determined final protocol is useful to assess materials' wear characteristics under the specified conditions. Several preliminary studies in this section provided information about the significant sources of experimental errors and facilitated the development of an experimental protocol which gives repeatable results. Accordingly this protocol was used for the final wear study discussed in the next chapter.

Chapter 8

***In vitro* Wear Resistance and the Strength of Three Restorative GICs**

8.1. Overview

In this chapter, the wear resistance and the physical strength of the three GICs (Fuji II Cap, Fuji IX, Fuji II LC, GC Cooperation, Tokyo, Japan) were tested. In applications such as the A.R.T. technique (Frencken *et al.*, 1994), GICs are the first choice since they possess beneficial properties such as fluoride release and chemical bonding to the tooth structure. In these cases, GICs are expected to serve as "long-term temporary" restorations, and thus the investigation of those two properties are important.

8.2 Introduction

Due to the advance of medical science, the number of medically compromised people such as organ transplant recipients and auto-immune disorder patients has increased. In these cases, it is often difficult or impossible for such patients to tolerate extensive and expensive dental treatments although they often suffer from extensive dental problems. Traditionally, the restorative procedures in these cases involved various modifications of zinc-oxide eugenol cement (ZOE). However, the stability and retention of ZOE is poor resulting in "patch-work" procedures (Leigh and Hunt, 1994). The recent introduction of GICs improved this situation due to their chemical adhesion to tooth structure and the therapeutic effects relating to fluoride. Furthermore, GICs are beneficial in dentition damaged with extensive erosion which is seen more with the increasing age of the population. The temporarization of occlusal surfaces in these cases with GICs is an ideal initial treatment because the irritation of the pulp is minimized and the remaining tooth structure is protected. GIC materials with maximum wear resistance and optimal strength are required to withstand occlusal forces for a relatively long term in such cases.

With progression of concepts of "remineralization", the technique called "Atraumatic Restorative Treatment (A.R.T.)" has been developed. Originally this technique was developed for use in less-industrialized countries and places where there are no facilities or financial resources to provide good oral health to the public. This technique involves excavating carious cavities in teeth using hand instruments and restoring them with adhesive filling materials such as GICs (WHO A.R.T. manual, 1994). Frencken *et al.* (1994) introduced the A.R.T. technique

in Africa and subsequently continued in Thailand with the support of WHO (FDI World, 1994). It is believed that this technique minimizes further caries development and helps remineralization in treated teeth which would otherwise not be treated. The restorations need to survive for a long period so that the prognosis for the teeth is optimised. Because it is a simple technique, large numbers of people can benefit from these treatments. Fuji IX (GC Cooperation, Tokyo, Japan) was especially developed for use with the A.R.T. technique and the manufacturer claims that this material is more resistant to occlusal forces.

The purposes of this study was to investigate the wear characteristics and the shear strength of the three commonly available GICs and to determine the most suitable GIC material for the "long term temporary" restorations .

8.3 Materials

Three kinds of GICs (Fuji II Cap, Fuji II LC and Fuji IX) were used in this chapter. Details of the materials used are shown in Chapter 7, Table 7.1.

8.4 Methods

8.4.1 Assessing the wear characteristics

Detailed materials and methods of these experiments have been described in the previous chapter (Chapter 7). The specimens made of three GIC materials (Fuji II Cap, Fuji II LC, Fuji IX) were rubbed against enamel specimens using the wear machine designed at the University of Adelaide (illustrated in Figure 8.1).

Enamel specimen

Two morphologically similar upper third molars collected from one patient were carefully selected. Each tooth showed a symmetric outline so that after the tooth was split longitudinally, four morphologically similar pieces were obtained. They were mounted in the way described previously and were rubbed against flat enamel for 15 minutes using the wear machine. By this method, enamel specimens with similar flat areas were created. Out of the four enamel specimens, the one which showed the least consistent surface size area was discarded. Each enamel specimen was used to wear against the five specimens made of the same material. Each time before wearing a new GIC specimen, the enamel surface was "roughened" by grinding against a flat enamel surface for 15 minutes on the wear machine to standardize the surface texture (see Section 7.5).

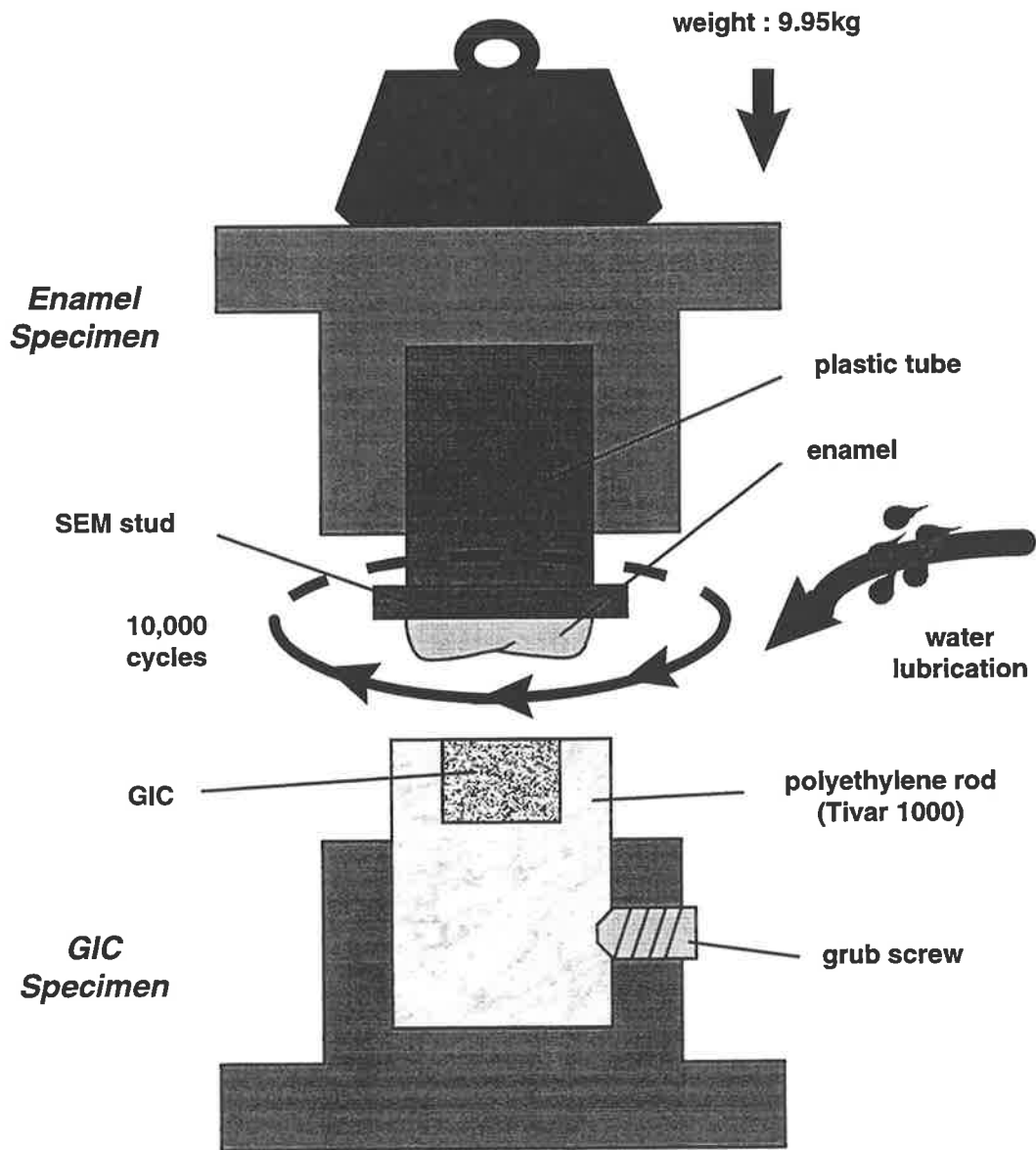


Figure 8.1 Operation of the Simulated Wear Machine.

GIC specimens

A Tivar 1000™ rod was sectioned into the fifteen standard shape pieces and the five specimens each of the three GIC materials (Fuji II Cap, Fuji IX and Fuji II LC) were prepared (details in Section 7.4.3). The samples were kept in water for one week at room temperature. Any excess was trimmed before measuring the original weight of GIC specimens. The specimen was worn against the enamel specimen at 9.95kg load at a speed of 80 cycles per minute for 10000 cycles. After every 1000 cycles, the GIC specimen was removed from the machine and wiped with a paper towel. It stood at room temperature for 3 minutes and was wiped again before measuring the weight. The wear indicated by the weight loss over the range between 2, 000 and 10,000 cycles. The results were analysed by a one way ANOVA test, a simple linear regression and a co-efficient of determination (STATVIEW 512+™ Version 1.0-Abacus Concept Inc).

8.4.2 Assessing the physical strengths

A total of ninety shear punch specimens were made in the manner previously described (see Section 4.3.2). Thirty specimens each of the three materials (Fuji II Cap, Fuji II LC, Fuji IX) making a total of ninety specimens were prepared. Ten specimens each of the materials were stored in water at room temperature for 2 hours, 1 week and 1 month, respectively. After storage, the physical strengths of each group were obtained using a shear punch test. Statistical analysis was performed using a two way ANOVA test (STATVIEW 512+™ Version 1.0-Abacus Concept Inc).

8.5 Results

8.5.1 The results of the wear resistance test

The results for accumulated weight change of the tested GICs over 10000 cycles are shown in Figure 8.2. The summary of this study is shown in Figure 8.3. The summaries of the results and the statistical analysis are described in Table 8.1 and Table 8.2. The wear rates of the three materials showed significant differences ($p < 0.01$). The wear rates between Fuji II Cap and Fuji IX, between Fuji II Cap and Fuji II LC showed significant differences ($p < 0.05$), but the wear rates between Fuji II LC and Fuji IX did not differ significantly ($p > 0.05$).

Combined with the fact that Fuji IX showed the most consistent results (higher co-efficient of determination) and the results of the water evaporation preliminary study (see details in 7.6) suggested that Fuji II LC showed more variation between specimens, it was concluded that Fuji IX showed the best wear resistance among the three GICs tested in this study, although the differences between Fuji II LC was not statistically significant ($p > 0.05$).

Table 8.1

The results of the statistical analysis of the wear rates (Fuji II Cap, Fuji IX and Fuji II LC)
(One way ANOVA test)

Source	df*	SS**	F ratio	
Between groups	2	1.87	16.253	p<0.0001
Within groups	117	6.736		
Total	119	8.607		

df*: Degree of freedom

SS**: Sum of Square

Table 8.2

The results of the statistical analysis of the wear characteristics of the three GICs
(simple linear regressions and co-efficient of determination)

Materials	Count*	Wear rate (mg/1000cycles)	SE**	r***	
Fuji II Cap	40	0.675	0.314	0.867	★ ☆
Fuji II LC	40	0.434	0.199	0.696	★
Fuji IX	40	0.392	0.186	0.883	☆

Count*: Number of the counts in each material between 2000 cycles and 10000 cycles (5 specimens each)

SE**: Standard error

r***: co-efficient of determination

☆☆ p < 0.05

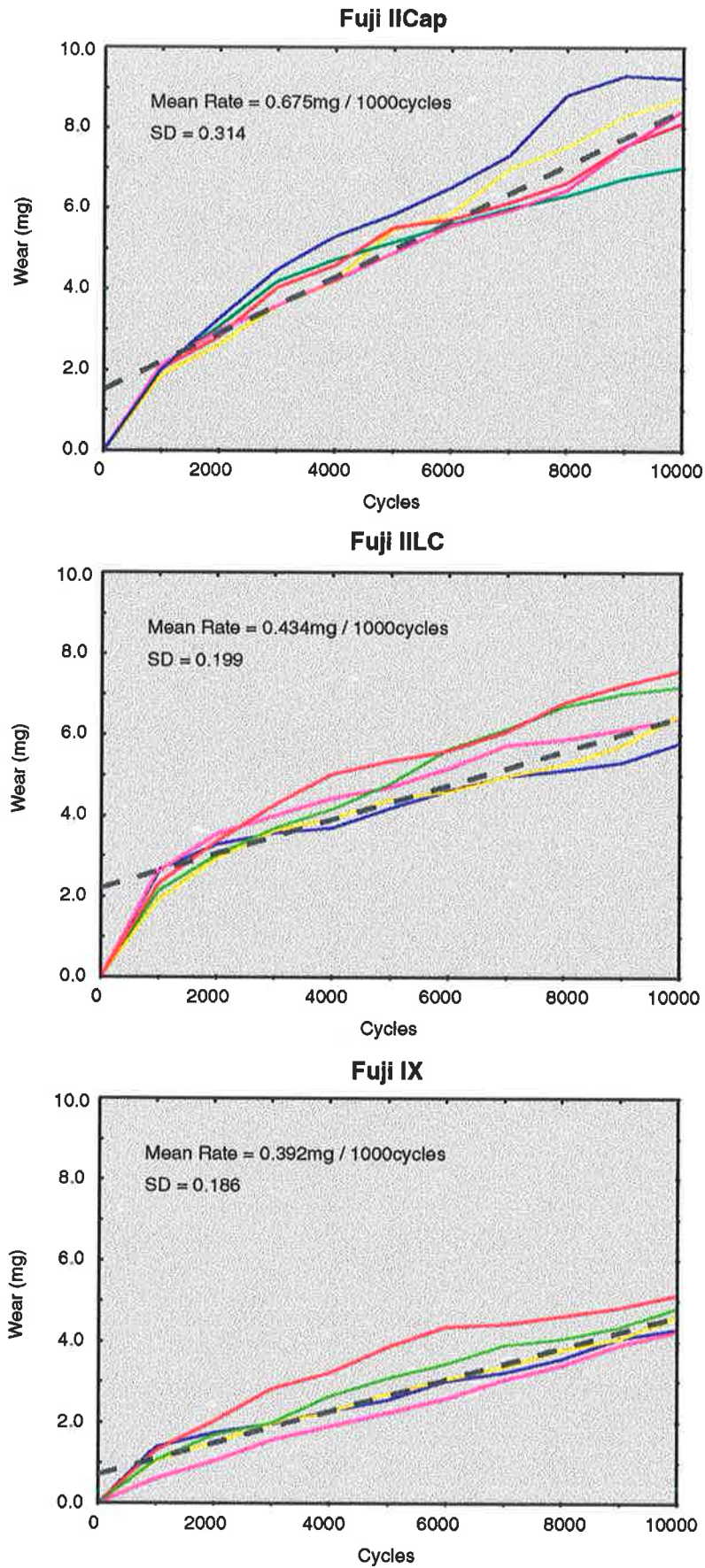


Figure 8.2 Results of Simulated Wear Test.

The graphs show the quantitative wear of the GIC specimens against enamel, versus the number of wear cycles. The coloured lines represent individual specimens while the dashed lines show the mean values between 2000 and 10000 cycles. The slope of these lines represents the mean wear rate.

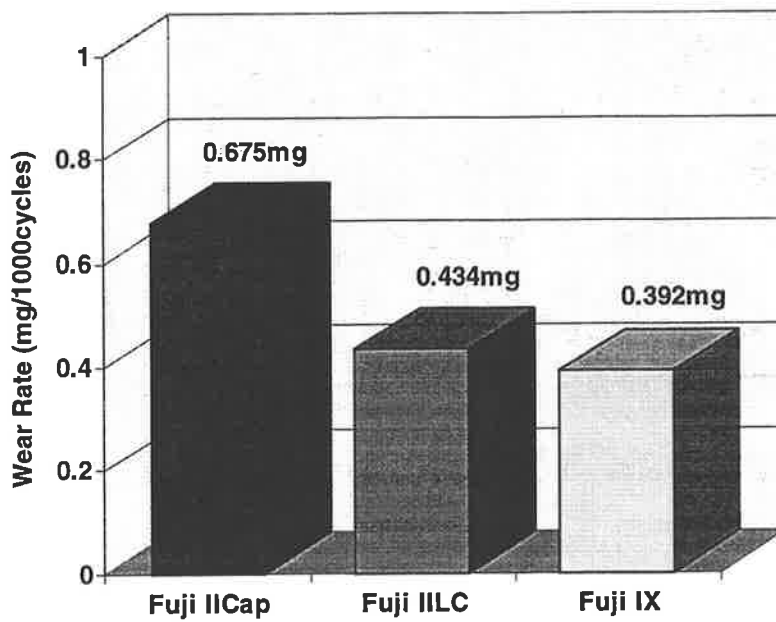


Figure 8.3 *GIC Wear Rate Summary.*

Fuji IICap showed the highest wear rate of the three materials and hence the lowest wear resistance.

8.5.2 The results of the shear punch test

The results of the shear punch tests are shown in Figure 8.4 and Table 8.3. The results of the statistical analysis are shown in Table 8.4.

Fuji II Cap specimens stored for two hours showed the lowest shear punch strengths and the one week old Fuji IX specimens recorded highest shear punch strengths. The shear punch strengths differed significantly depending on storage periods ($p < 0.01$). The strengths of all specimens increased over one week, but there were no obvious changes in between one week and one month except for Fuji IX which recorded the highest value after one week and a lower value after one month. Of the 2 hour specimens, the Fuji II Cap specimens showed low strength compared with the Fuji II LC and Fuji IX specimens. These two materials stored for two hours showed similar strengths. However, the Fuji II Cap specimens increased to double the strength after one week. The strengths of the three materials stored for one month showed no significant differences ($p > 0.05$). Of the one month specimens, the Fuji II Cap specimens showed slightly higher values than the other two materials.

Table 8.3

The results of the shear punch tests of this study. The sample size, mean strengths and standard deviations (SD) of the each series of the specimens. (Two way ANOVA)

Condition*	Material	N**	Mean strength (MPa)	SD
I	Fuji II Cap	10	31.49	2.69
II	Fuji II Cap	10	67.68	5.60
III	Fuji II Cap	10	69.23	6.52
I	Fuji II LC	10	43.51	4.88
II	Fuji II LC	9	66.33	5.63
III	Fuji II LC	10	65.27	5.29
I	Fuji IX	10	45.18	2.81
II	Fuji IX	9	75.64	4.34
III	Fuji IX	10	67.00	5.80

Condition*: The specimens of Group I, II and III were tested in 2 hours, 1 week and 1 month (after mixing) respectively.

N*: Number of the specimens

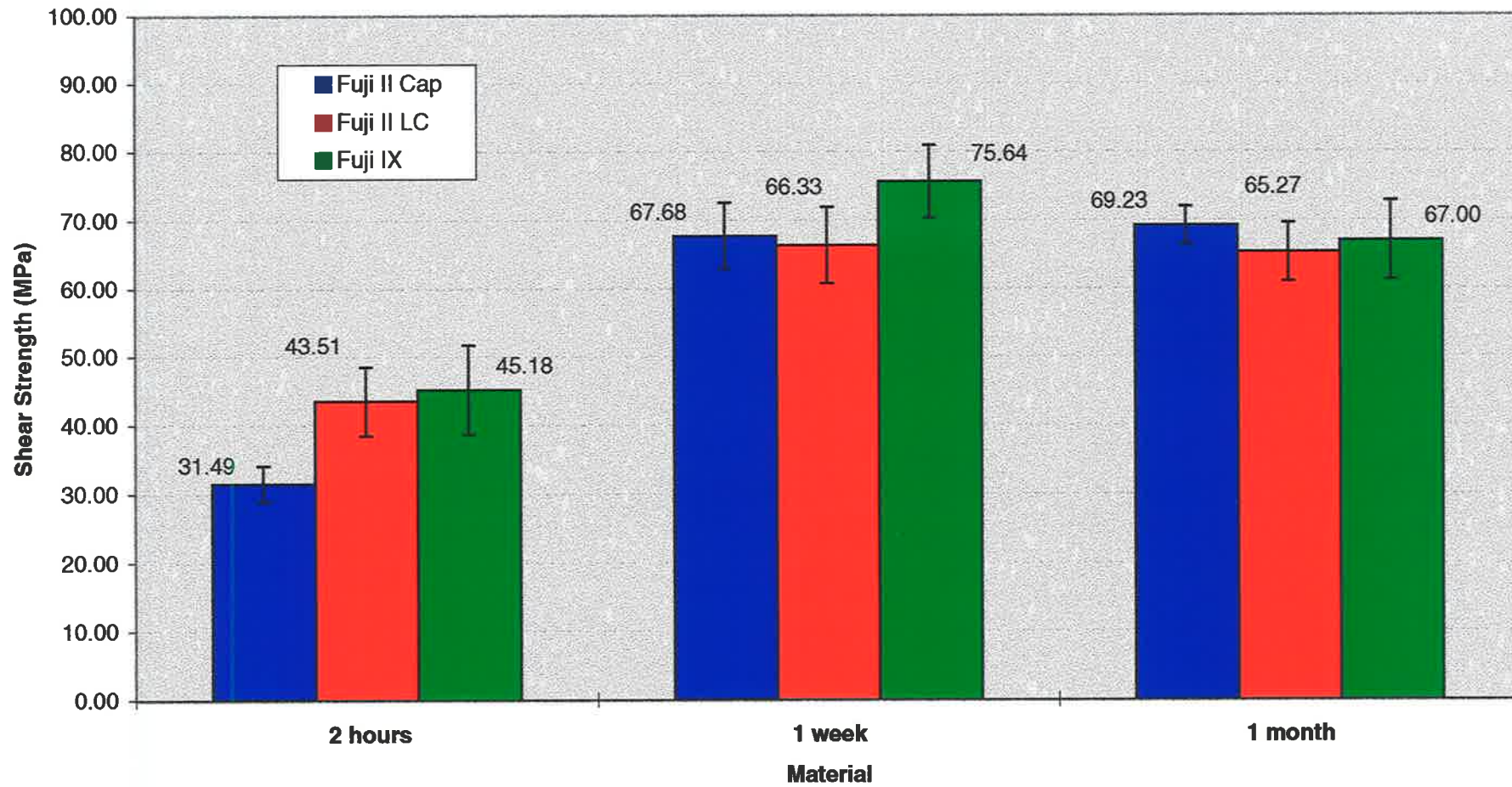


Figure 8.4

Maturation of three kinds of GICs (Fuji II Cap, Fuji II LC, Fuji IX)

The mean shear strength of three GICs stored in water for 2 hours , one week and one month.

* Error bars represent the standard deviation.

Table 8.4

The results of the statistical analysis of the shear punch tests of the three GICs.
(Two way ANOVA test)

Source	df*	SS**	F-test	P value
Materials (A)	2	633.57	12.68	<0.0001
Condition (B)	2	16066.01	321.50	<0.0001
AB interaction	4	1039.15	10.40	<0.0001
Error	79	1973.90		

df*: Degree of freedom

SS**: Sum of Square

8.7 Discussion

It is generally accepted that the strength of GICs increases over time (Wilson *et al.*, 1979). However, there is controversy regarding the duration of "maturation" effects. For example, Williams and Billington (1989, 1991) found compressive strengths peaked around 50 days, and in the other study, four-point flexural strengths of several cements were found the peak around 24 hours after mixing (Pearson and Atkinson, 1991). Suzuki *et al.* (1995) found one year old Fuji IX specimens showed 27.5% higher compressive strength than that of the first day. The study by Ishihara and Tosaki (1993) using Fuji II and Fuji II LC showed similar compressive strengths in one week and one year aged specimens.

According to the shear punch data in this section, all the materials increased in strength between 2 hours and one week, but did not show a trend for continuous increase between one week and one month. The Fuji II LC and Fuji IX specimens even showed slightly lower strengths in the one month old specimens than the one week old specimens. An interesting finding was that the Fuji IX specimens showed similar strengths to the Fuji II LC specimens after 2 hours although a higher strength was expected in the Fuji II LC specimens due to the resin component and its snap setting property. Moreover, in the one week old specimens, Fuji II LC showed similar strength to Fuji II Cap. These results suggest that Fuji II LC did not show physical strength superior to the other conventional GICs. However, these are the results observed over a limited time frame and further study is necessary to accurately describe the relationship between strength and time.

In general, clinical wear of a material is a combination of several types of wear (McCabe and Smith, 1981). Material losses in occlusal contact areas and in contact-free areas are considered to be caused by different wear mechanisms. For example, in CRs and GICs, in contact free areas, wear is due to microabrasion of the matrix, protrusion of filler particles and dislocation of fillers. Occlusal wear is perhaps due to deformation of the surfaces and microcracks generated within the material, resulting in a crack running parallel to the surface (Bauer *et al.*, 1996). This kind of mechanism is difficult to comprehend as the applied stresses vary and fatigue cracks may grow in the subsurface area (Wu *et al.*, 1984; Braem *et al.*, 1986). In fact, this also indicates that what often is described as wear has probably an identical underlying primary mechanism to that seen in marginal breakdown or chipping or cracks under cyclic loading (Braem *et al.*, 1995). Naturally the expression of this damage differs depending on the type of material tested (Goldman, 1985; Tyas, 1990). However, this kind of microcrack mechanism is likely to occur in GIC wear because GICs are brittle materials. In this aspect, perhaps the physical strengths and wear resistance may correlate in the case of GICs. However, in this study, the results of the wear rate and the strength were not entirely correlated. For example, the Fuji II Cap specimens (one week old specimens) showed the lowest wear resistance of the three materials (significant at 95% confidence level) which was not expected from the shear punch results where Fuji II Cap showed slightly higher strength compared to Fuji II LC (one week old specimens). This suggested that the wear process is not only related to strength but also to more complicated processes which might include the bond strength of the particle and matrix, the hardness of particles and brittleness.

Because of the limited thickness of an enamel layer, it was not possible to increase the sample size beyond five under the protocol for these wear experiment. This was not because of the wear of enamel against the GIC but due to the repeated grinding process against opposing enamel to standardize the enamel surface which reduced the thickness of enamel.

8.7 Conclusion

The ranking of wear resistance based on the data from this study was Fuji IX > Fuji II LC > Fuji II Cap. However, it should be noted that these results were obtained under the specified experimental conditions. For example, it should be noted the areas of the enamel specimens were not strictly controlled in this study. Also, the hardness of enamel may change as it wears close to the DEJ. While this might be taken as an indication of the performance of these materials, the final assessments should be based on clinical studies. Clinical wear studies by Roulet and Walti (1984), for example, have shown that chemical dissolution was a significant

factor in the disintegration of cermet restorations and other complex conditions which can not be simulated in the *in vitro* situation may influence the results.

Some properties of three GIC materials including shear punch strengths and wear characteristics were examined to identify suitable materials for "long-term temporary" restorations. Under the experimental conditions considered, Fuji IX appeared to have the most appropriate characteristics for 'long-term temporary' restorations. However, final evaluation should be obtained from future controlled clinical studies.

Chapter 9

Discussion

The shear punch test and the simulated wear test which have been extensively discussed in this study appeared to be reliable to assess some aspects of the properties of dental restorative materials. A large part of this study was devoted to a series of preliminary studies to establish the protocols because neither test are ISO standard tests and limited information was available. For the wear study, several problems such as the amount of water evaporation and the size of the enamel specimen have been well controlled in this study. The sample size could be extended in future experiments to get more conclusive results.

As in all *in vitro* studies, the specific experimental conditions of this study might have affected the results and the material may behave differently in the clinical situation. For example, curing, storage and testing were all carried out at ambient room temperature in this study. Also particular chemical conditions may influence the *in vivo* performance of the GICs. For example, lactic acid which is produced during plaque metabolism is known to attack dental cements (Crisp *et al.*, 1980b).

Many aspects of the properties of the GICs have been discussed in the earlier sections. Taken together the results of these studies suggest that compared with earlier materials, RM GICs did not appear to show significant improvement in the tested properties. Newly developed RM GICs were earlier reported to show improved clinical performance, handling, and range of usefulness (Mitra, 1989). The generally improved appearance, physical properties, and adhesion to tooth structure of the RM GICs were believed to offer the potential for superior clinical results by many clinicians (Smales and Koutsikas, 1995). However, the results of this study suggested that the "superior" physical properties are merely reflecting a quick set of the RM GICs at the early setting stage (which is still advantageous to the early moisture sensitivity), and not an improvement in the strength of the materials themselves.

Research into the strengths of GICs could also improve of the bond strength between GIC and tooth structure as there is a reason to believe that reported bond strengths may be artificially low because the materials commonly fail cohesively rather than at the tooth-material interface (Mount, 1991). GICs are known as the materials which chemically adhere to tooth structure and therefore improvements in the strengths of these materials offer significant improvement in the

clinical performance.

The effects of the "maturation" of GICs were discussed in several sections in this study. While "maturation" could be a favorable characteristic of GICs, the materials may also develop other changes. It should be emphasized that strength is not the only parameter to judge the superiority of the materials. For example, RM GICs may absorb more water than conventional GICs in the long term as HEMA is the resin component which polymerizes during the light activation process and is hydrophilic (Nicholson *et al.*, 1992). The results of this study suggested that RM GICs showed a trend to resist crack formation in dehydrated conditions, but did show water evaporation from the specimens similar to that seen in conventional GICs.

There are several reports showing the hygroscopic characteristics of RM GICs. Hinoura *et al.* (1993) examined the volumetric changes of RM GIC and found a linear decrease up to 5 minutes after the restoration was placed followed by expansion up to 6 hours when the specimens were kept in water. Similar finding was reported by Irie *et al.* (1992) studied the dimensional changes in several RM GICs using Teflon cavities. They concluded that the setting shrinkage of RM GICs lead to marginal gaps. They observed that the marginal gaps decreased after storage in water for one day. They concluded that the cause of the dimensional change was due to the hygroscopic expansion. More recently, a study of the water content of some materials was carried out by Small *et al.* (1996) using radioactive materials and a scintillation counter. They examined patterns of water uptake of RM GICs (Fuji II LC), conventional GICs (Fuji II), CR (Herculite) and polyacid-modified CR (Dyract). They found RM GICs showed highest (8.9% wv) water uptake while conventional GIC and CR showed the lower water uptake (5.4% wv and 1.23% wv, respectively). Another study by Cattani-Lorente *et al.* (1996) found that RM GIC (Fuji II LC) showed approximately four times as much water sorption as conventional GIC (Fuji II) after 24 hours. This result may suggest that RM GICs might be more prone to discolor in the long term. However, there is little information regarding the clinical behaviour of RM GICs in the literature. Obviously, further research in this area is mandatory.

Ideally, it is very important to assess all aspects of the properties of dental materials. However, many researchers created different experimental conditions and as a result the data obtained from those studies are not comparable and none of the tests entirely predicts clinical performance. Based on the results of this study the materials were ranked. It should be emphasized that this ranking provided information based on one aspect of the properties of the materials. Bream *et al.* (1995) stated that any ranking made of materials based on one property is inadequate since the ranking is altered if another property is taken into account. However,

some information regarding the wear resistance and physical strengths of the material was useful.

Whatever information is available, a final decision about materials should be made from the clinical results and long term observations of newly developed materials such as RM GICs are important. Further research should be encouraged to improve the physical properties of GICs.

Chapter 10

Conclusion

Modern dentistry is changing. There was an age when dentistry focused entirely on the mechanical properties of dental materials. Up to the 1950's the focus of research and usage of a dental material was judged by its physical and mechanical properties. However, since more recent investigations and experiments have expanded the knowledge of tooth structure and adhesive materials, it is now possible to restore teeth with a minimum of damage to tooth structure. With the development of this "minimum intervention" concept in restorative dentistry, adhesive materials have played an important role. In particular, the direct filling composite resins with improved properties, and the evolution of the acid etch technique and dentine bonding systems have been significant.

Higher demand for aesthetics has also necessitated alternatives to currently used amalgam alloys. In addition to this, the use of mercury in amalgam has created constant controversy and public pressure to find alternative has arisen (Roulet and Losche, 1994). While there is an opinion that amalgam should be abandoned as a restorative treatment for caries and that CRs or RM GICs are more suitable alternative materials (Simmons, 1995a), this view has been criticized (Mjor, 1995) as amalgam still appears to show superior physical properties especially in the areas where occlusal forces are relatively high. However, in less stress bearing situations, if current trends continue, these 'aesthetic amalgam substitutes' such as GICs and CRs will probably be more accepted than cast gold or porcelain restorations (Burgersdijk *et al.*, 1991, Roulet and Losche, 1994).

It is a fact that CRs are not always the material of choice due to disadvantages such as polymerization shrinkage and lack of biocompatibility. On the other hand, GICs have been developed as materials which show unique properties more compatible with tooth structure. Not only do GICs adhere chemically to tooth structure, but also fluoride release contributes to additional benefits. However, the results of this study suggested that currently available GICs including RM GICs showed significantly lower strengths compared with CRs. This data suggested that the applications of these materials in possible load bearing areas should be carefully assessed. In such cases GICs must be handled adequately to obtain optimal results.

Another change influencing modern dentistry is the increasing life expectancy of populations

throughout the world. Especially after the year 2010, this aging tendency will be accelerated, and it is expected that people 60 years of age and over will represent approximately 25% of the total population by the year 2035 (Kalk *et al.*, 1992). Naturally, there is a tendency for more people to preserve their natural dentition into old age (Linden, 1985; Katz and Gustavsen, 1986). They tend to have different restorative problems such as root caries and excessively worn dentition (Mair, 1992) and the treatment of these cases are often complicated (Crothers, 1992). The applications of GICs are increasing in these cases and useful information about the properties of GICs were obtained in this study.

In any case, the applications of dental materials should be carefully judged because no dental material is perfect and each material has different properties. The speed of the development of new dental materials is accelerated more than ever and it is becoming more difficult to follow all the innovation and new information. Under these circumstances, it is also very difficult to obtain information about long term behaviour. Further research to investigate the long term clinical behavior of these materials is mandatory. At the same time, further innovation and research into GICs with improved physical properties is required to spread the horizon of the application of GICs. The ultimate goal is to develop a restorative material which in all aspects possesses similar properties to tooth structure as much as possible.

On many occasions, we choose the most appropriate materials which hopefully enables us to obtain optimal results in clinical situations. An ability to make a fair and precise judgment about the materials is needed more than ever. Under these circumstances, it is important to judge non-biased results. The two *in vitro* tests discussed in this report, namely the shear punch test and the wear test appeared to successfully supply useful clinical information about the appropriate use of these materials.

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