

## THE PERFORMANCE

# OF MATERIALS AS A TEMPORARY ENDODONTIC SEAL

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## **DECLARATION**

This report is submitted as partial fulfilment for the Degree of Dental Surgery.

The research is of original material and work and has not been previously submitted for any degree.

Where the research findings of other workers is described, mentioned or discussed, due reference has been made and included in the Bibliography.

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Paul S. Heijkoop

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## **DEDICATION**

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I dedicate this thesis to my dearest friend and wife Katherine. For all those long and frustrating hours keeping me in body and soul while I spent more time with con! than with you.

## ABSTRACT

Endodontic therapy usually involves treatment over several appointments necessitating temporary restoration of the coronal access cavity. This temporary access cavity seal is important in attaining and maintaining a sterile root canal system.

The dubious performance of traditionally used materials particularly in the long term, is well documented in the literature, where most studies have only been carried out in virgin teeth.

It was the aim of this study to assess the suitability of dental materials not customarily used as restorative materials, and some epoxy resin based industrial materials, as long term temporary endodontic coronal sealing agents, in conjunction with materials currently in use.

This was in order to indicate which of a series of materials might show promise for further investigation and modification, rather than to develop a new material.

Materials tested included: Cavit, encapsulated IRM, TERM, encapsulated Ketacbond, Genesis, Ramitec, Impregum F, Permadyne, 10 minute setting E-POX-E putty, 2 hour setting E-POX-E putty, and an Araldite aluminium oxide mixture with a powder liquid ratio of 2:1. setting prior to a patient leaving the operatory.

Microleakage studies with cavities in endodontically prepared teeth, amalgam composite resin and Ketac Silver and 100 thermocycles between baths of 1% methylene blue dye at 4 °C and 60 °C showed IRM to leak extensively in tooth and amalgam and Ketac-bond to leak extensively in tooth leading to discontinuation of investigation on these materials. TERM sealed well in all substrates other than amalgam. All other test materials sealed adequately in all substrates.

Cavit and Genesis showed most of their linear expansion to take place in the first 24 hours when tested in distilled water at 37 °C at 24 hours and 14 days. All other materials except 2 hour E-POX-E putty showed a significant increase in expansion to 14 days.

In vitro wear investigations on a brushing machine using Zircate paste as an abrasive showed Permadyne to have the least wear, but this was not significantly less than for the other polyether materials, 10 minute E-POX-E putty and Araldite. TERM showed significantly less wear than Cavit which showed the greatest wear of all materials tested.

Ledermix paste and Pulpdent paste had no effect on the hardness of set materials. Ledermix paste induced surface staining of all materials.

Of the traditional temporary materials tested only Cavit sealed satisfactorily in all substrates but showed poor wear resistance. The polyether and epoxy resin based materials performed well in comparison to the traditional materials and subject to further usage and biocompatability testing may be suitable as temporary endodontic access cavity seals.

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### PREFACE

This work was carried out to assess the possible suitability of several dental materials, and some epoxy resin based industrial materials, for use as a long term temporary endodontic coronal sealing agent. Various <u>in vitro</u> tests were carried out assessing the performance of these materials as compared to three control materials, Cavit<sup>1</sup>, TERM <sup>2</sup>, and IRM <sup>3</sup>, which are commonly used as temporary endodontic coronal seals.

Tests considered most relevant to successful performance of a long term endodontic temporary coronal seal were carried out in a particular sequence and a material was excluded from further study if it failed to reach an acceptable standard with a particular test.

It was not the aim of this study to develop a new material but rather to indicate which of a series of materials might show promise for further investigation and modification.

This study was designed as a sieve prior to any necessary toxicity studies (for nondental materials) which would have to be addressed in a further project.

<sup>&</sup>lt;sup>1</sup> Cavit, ESPE, Gmbh & Co., West Germany.

<sup>&</sup>lt;sup>2</sup> TERM, Caulk/dentsply International Inc., Milford, Del., USA.

<sup>&</sup>lt;sup>3</sup> IRM, Caulk/Dentsply International Inc., Milford, Del., USA.

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## **INTRODUCTION**

The endodontic treatment of teeth with irreversible pulpal or periapical pathology has as its prime objective the repair of the connective tissues surrounding the tooth root. Treatment aims essentially at the elimination of the microbiological causative agents and the subsequent placement of inert materials to seal the canal system from re-entry of microorganisms. It has been shown (Byström, Claesson and Sundqvist, 1985) that the elimination of microorganisms cannot be achieved in one visit by chemo-mechanical preparation alone and intracanal medication on at least one occasion is required to eliminate all bacteria. Hence treatment is generally carried out over at least two appointments, the interval between appointments being determined by the nature of the treatment and the intracanal medication used. The duration of this interval is usually in the order of one to two weeks, but it may extend over a period of several months for personal reasons such as illness or where complex treatment such as apexification is involved.

During the course of endodontic therapy it is important that contamination of the root canal system is prevented. A temporary restorative material is used to seal the root canal system from the oral environment and prevent the ingress of organisms, their toxins or material which may enhance the growth of existing bacteria within the root canal. An antigenic material may have a molecular weight as low as 1000 (Roitt, 1977) and although the smallest size particle capable of eliciting a periapical inflammatory

response is unknown, the seal provided should be such that the ingress of these particles is prevented.

To date several materials have been used for this purpose but no material has been developed which adequately fulfils ideal performance criteria.

(Grossman, 1981) has listed the following criteria for a temporary endodontic filling material which:

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- (1) should be impervious to bacteria and to fluids of the mouth;
  - (2) should hermetically seal the cavity peripherally;
  - (3) should not cause pressure upon the dressing during (it's) insertion;
  - (4) should harden within a few minutes after insertion in the cavity;
  - (5) should withstand the force of mastication;
  - (6) should be easy to manipulate;
  - (7) should be easy to remove;
  - (8) should harmonize with the colour of tooth structure".

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### LITERATURE REVIEW

#### 2.1 MATERIALS COMMONLY USED AS A TEMPORARY ENDODONTIC SEAL

Currently there is no one material which is accepted as the temporary sealing material for endodontic use in all clinical situations. This has resulted from the different situations which may arise during the course of the treatment. The market place also provides a vast range of materials which although being of the same class may exhibit some variation in properties. These factors combined with conflicting reports from investigators leads to the general use of several different materials by clinicians.

Generally the category of materials known as dental cements have been used as temporary restoratives. These may be classified

"according to the matrix-forming species:

- (1) phosphate-bonded,
- (2) phenolate-bonded,
- (3) carboxylate-bond, and
- (4) methacrylate (resin)-bonded" (Smith, 1983).

Although methacrylate (resin)-bonded cements are not used as temporary restoratives, composite resins have been used to effect a temporary seal during endodontic therapy. There are also several other proprietary products specifically marketed for the temporary sealing of endodontic access cavities which do not fall into the categories of

cements listed above. Historically gutta-percha stopping has been used but this practice is no longer followed because of leakage.

Some commonly use materials are listed below.

1. Zinc phosphate cement,

2. zinc oxide-eugenol cement,

3. Reinforced zinc oxide-eugenol cement,

4. zinc polycarboxylate cement,

5. glass-ionomer cement,

6. composite resin,

and 7. calcium sulphate/zinc oxide-zinc sulphate based materials.

Of the materials listed above the most commonly used materials are IRM (Intermediate Restorative Material) a resin reinforced zinc oxide-eugenol, Cavit, a calcium sulphate/ zinc oxide-zinc sulphate based material and a new material, TERM (Temporary Endodontic Restorative Material), a light cured resin based system.

#### 2.2 MICROLEAKAGE

#### 2.2.1 Introduction

Of primary importance when carrying out endodontic therapy is the attainment and maintenance of sterility in the root canal system. The junctions between tooth and restorative material are " dynamic microcrevices which contain a busy traffic of ions and molecules" (Myers, 1966). With this factor in mind there has been emphasis in the literature on the ability of restorative materials to effect a seal at the restoration-tooth interface preventing the ingress of organisms or their products.

Several methodologies have been developed to study the sealing ability of the various materials used as temporary endodontic filling materials <u>in vitro</u>; however the extrapolation of these results to the clinical situation where materials may be manipulated in a less than ideal manner is often difficult and may be misleading. To date there are only a few relevant <u>in vivo</u> studies which have been carried out (vide infra).

#### 2.2.2 Methods of assessing microleakage

Several methods have been devised to assess microleakage at the interface between tooth and restorative material ranging from simple inexpensive techniques to complex techniques beyond the reach of most researchers.

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#### Methods of investigation:

- 1. Visualization.
- 2. Air pressure.
- 3. Electrical conductivity.
- 4. Dye penetration.
- 5. Radioactive isotopes with autoradiography.
- 6. Bacterial penetration.
- 7. Neutron activation analysis.
- 8. Radiochemical diffusion.
- 9. Reverse radioactive absorption.

#### 2.2.2.1 Visualization

Visualization is the oldest method used to study microleakage and was first described in 1895 (Kidd, 1976).

The development of the scanning electron microscope has enabled the use of direct visualization of the interface between tooth and restorative material with excellent depth of focus and magnification. This method measures the size of the microcrevice between the tooth and restorative material. The potential for artefacts to develop during specimen preparation detract from its use (Going, 1972), however in order to minimize this problem replication techniques have been developed (Grundy, 1971; Pameijer and Stallard, 1973).

#### 2.2.2.2 Air pressure

Air pressure was first introduced for the assessment of the marginal seal of fillings in 1912 by Harper. Since this time it has modified by varous investigators (Knappwost, 1951; Fiasconaro and Sherman, 1952; Pickard and Gayford, 1965; Granath and Svensson, 1970). It is a quantitative technique for use <u>in vitro</u> with the advantage that it does not result in the destruction of the specimen thus allowing for assessment over an extended time period (Shortall, 1982).

#### 2.2.2.3 Electrical conductivity

Another non-destructive and quantitative technique for the measurement of marginal leakage was developed by Jacobsen and von Fraunhofer in 1975. This conductimetric technique measured the change in dimension of the microspace between tooth and restoration using an electrochemical cell. In review Shortall (1982), felt that this method lacked realism as the experiments were carried out in glass and that cavities <u>in vivo</u> have rough rather than smooth surfaces, are moist and have varying surface energies which affect their wettability and the intrusion of any chemical agent.

More recently, Delivanis and Chapman (1982), when comparing the reliability of techniques for measuring microleakage in retrograde seals found that an electrochemical method employing zero resistance ammetry showed good correlation with both dye penetration and autoradiographic methods.

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The technique of using organic dyes to measure the leakage of restorations is one of the oldest. It is both simple and inexpensive and is thus available to most investigators. Although several dyes have been used, the use of fluorescent dye (fluorescein) was found to be particularly useful because of the ease of detection in dilute concentrations and sensitivity to ultraviolet light. Dyes are easy to photograph and permit reproducible results (Going, 1972). Fluoresein is nontoxic and has been used <u>in vivo</u> to study microleakage in the teeth of golden Syrian hamsters and in humans (Loiselle et al., 1969).

The problem with dye penetration studies is that the results are not quantitative thus preventing comparison between studies. It is also necessary to destroy the specimen preventing continuous monitoring of the leakage (Crisp and Wilson, 1980).

An interesting method employing compressed air to force dye through a margin and quantify the volume movement of the dye using an entrapped air bubble in a capillary tube has been developed by Derkson et al. (1986). This has allowed quantitative and non destructive monitoring of dye leakage.

#### 2.2.2.5 Radioactive isotopes

The use of radioactive isotopes to measure leakage was introduced by Armstrong and Simon in 1951. Radioactive salts are used as tracers to penetrate the tooth-filling interface. Autoradiographs are then taken of sectioned specimens to detect the presence of an isotope and thus the depth of penetration.

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Going et al (1960), showed isotopes to have an inherent ability to penetrate more deeply than dyes due to their smaller molecular size. They also showed that the autoradiographic technique could detect minute amounts of isotope which otherwise could not be detected visually, indicating that this method was more sensitive than dye methodologies. It has been questioned as to whether this is an advantage as a nonleaking system could not be found, and there is doubt as to whether it is capable of discerning a nonleaking system (O'Brien et al., 1968).

In a study to determine whether ionic exchange and chemical reactivity influenced the degree of penetration it was found that these factors as well as the chemical nature of the filling material influenced the depth of marginal penetration. In addition <sup>35</sup>S and <sup>45</sup>Ca showed selective and deep penetration into marginal defects and produced the clearest and sharpest autoradiographs (Going et al., 1960b).

Disputing these findings Kapsimalis, Evans, and Tuckerman (1965), and Kapsimalis and Evans (1966), in assessing the sealing of endodontic filling materials found different isotope usage did not affect the leakage pattern seen.

<sup>45</sup>Ca is the most widely used isotope in leakage studies. It is a weak beta emitter and thus produces sharp autoradiographs, however ion exchange with calcium of the apatite crystals in tooth can occur and will interfere with the recognition of the true leakage pattern (Delivanis and Chapman, 1982).

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In contrast to the results of Going et al. (1960), a comparison of methods used to assess marginal leakage of root canal fillings found methaline blue dye to penetrate further up the canals than the isotope tracers,  ${}^{45}Ca$ ,  ${}^{14}C$  labelled urea and  ${}^{125}I$  labelled albumin. Of the isotopes  ${}^{14}C$  labelled urea penetrated furthest (Matloff et al, 1982).

The technique of autoradiography itself has several variables affecting the resolution of the film such as the energy of the isotope used, (lower energy isotopes give better resolution), the distance from the specimen (source) to the reactive particles in the emulsion, and the exposure time (Fischer and Werner, 1971 and Rogers, 1969). These results are therefore qualitative only, with comparison between studies being extremely difficult.

#### 2.2.2.6 Bacteria

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Fraser's 1929 study is quoted as being the earliest to investigate bacterial penetration around restorations in glass (Shortall, 1982). Since then there have been numerous studies looking at bacterial penetration around restorations.

Several different approaches have been taken such as observing artificial secondary caries production (Ellis and Brown, 1967), and microscopic examination of cavities <u>in vivo</u> (Lamers et al., 1980).

These studies were more clinically orientated but as with many other penetration techniques were not quantitative (Going, 1972).

It must also be remembered that some restorative materials possess antibacterial properties (Tobias et al., 1985).

#### 2.2.2.7 Neutron activation analysis

This is reported as the only <u>in vivo</u> method of studying diffusion around dental restorations that yields a quantitative result (Going et al., 1968).

The technique as described by Going (1972), involves the isolation of a functional vital tooth with a latex isolator developed for the purpose. The isolator is injected with a manganese solution which will then penetrate any microcrevice present. After extraction the teeth are placed in the core of a nuclear reactor along with internal standards to convert the manganese to a radioactive state. The measurement of gamma-ray emission can then be calculated and expressed as the uptake of micrograms of manganese per tooth.

This is obviously a very complex and expensive technique and is beyond the reach of most researchers thus limiting its use in routine microleakage studies.

#### 2.2.2.8 Radiochemical diffusion

This is a more recently developed technique using radioactive ions in a diffusion cell model which can give a quantitative measurement of ion diffusion across a section of filled tooth (Crisp and Wilson, 1980). Although the method cannot be applied <u>in vivo</u> it is quantitative and may be suitable for continuous monitoring as the specimen does not need to be destroyed for measurement as is necessary in many of the methods described above.

#### 2.2.2.9 Reverse radioactive absorption

This alternative technique uses the detection of radioactive material (tritiated leucine) to quantify micoleakage. It involves the placement of the tritiated leucine in the floor of the cavity below the restoration. The specimen is then washed and placed in a solution of non-radioactive leucine which is then monitored by measuring successive aliquots for radioactivity (Vasudev et al., 1981).

#### 2.3 FACTORS AFFECTING MICROLEAKAGE

#### 2.3.1 Thermal cycling

The effect of temperature on the size of the space between tooth and filling was first shown to be of importance by Nelsen, Wolcott and Paffenbarger in 1952. They demonstated that on heating a restored tooth which had been cooled to 9 °C in a bath of ice water that drops of water were extruded from the margins of the restorations. This led to the development of the theory that the difference in the coefficient of thermal expansion of tooth, and the restorative material, resulted in marginal percolation of oral fluids.

Bauer and Henson in a review article in 1984 suggested that with temperature variation the difference in coefficient of thermal expansion contributes 90% to the fluid exchange during thermal cycling.

The clinical significance of thermalcycling has been questioned by Glyn Jones et al. (1978), who found that cycling at clinically realistic temperatures did not affect microleakage around composite restorations. They stated that <u>in vitro</u> testing of microleakage assessed the initial seal produced and not necessarily the long term sealability.

Harper et al. (1980), demonstrated that the temperature change under unfilled resins during eating and drinking was relatively small because the heat diffused slowly through these materials. This supports the view that in resin based restorative materials other factors such as modulus of elasticity and thermal diffusivity might be of more importance in cavity seals than the coefficient of thermal expansion. Whether this is the case with other restorative materials has yet to be determined.

### 2.3.2 Mechanical cycling

When viewed under a measuring microscope it has been demonstrated that axial loading of restored teeth results in a permanent or transitory gap formation indicating a risk of marginal percolation (Jorgensen, 1976).

Qvist in an <u>in vivo</u> study in 1983 demonstrated a significantly greater degree of bacterial penetration indicative of marginal leakage in class V resin restorations in teeth with an antagonist as compared to teeth without an antagonist. In that study cavity margins were acid etched but no unfilled resin bond was used. These findings were contrary to those of Asmussen (1977), who demonstrated the prevention of gap formation <u>in vitro</u> without unfilled resin linings even when thermocycling was carried out. Qvist concluded that while they are not the only cause of marginal leakage, masticatory forces play a major role. He stated that "dissolution or aging of the composite material, together with functional abrasion and thermal stress of the restorations, is without significant influence on the marginal adaption of composite restorations in acid-etched cavities".

#### 2.3.3 Setting contraction

Many dental materials undergo dimensional changes on setting. Of particular

importance in microleakage is setting contraction which may lead to the material pulling away from the cavity wall. Composite resin materials have as one of their major problems polymerization contraction (1.5% by volume approximately Phillips, 1982). The forces generated by this contraction on polymerization may exceed the bond strengths of currently available dentine bonding agents.

#### 2.3.4 Durability

The ability of a material to remain in place during the required period is obviously a key factor in preventing contamination of the root canal system. The various physical properties of materials used for the temporary endodontic seal will be discussed in a later chapter.

#### 2.3.5 Manipulation

The technique used to mix and place a temporary restoration could affect its performance and sealability. Chohayeb and Bassiouny (1985), indicated that single paste materials performed better than those requiring mixing and suggested that the ease of handling may be an important factor. Marosky et al. (1977), also felt that the manipulative factors contributed to the better sealing ability of single paste materials in their study.

#### 2.4 MICROLEAKAGE STUDIES

#### 2.4.1 dye penetration studies

There have been several studies conducted using dye penetration as a means of trying to identify which of the materials investigated provides the best seal under the conditions of the particular test used. Because of the various methodologies employed comparisons between studies is extremely difficult, and as most of these studies have been conducted <u>in vitro</u> and extrapolation to the clinical setting must be limited to the broadest trends only.

Parris et al. (1960, 1964), investigated a range of materials at room temperature and after thermocycling (4 °C - 60 °C) using analine blue dye. Materials assessed included gutta-percha materials, zinc phosphate cements, an amalgam, a zinc oxide eugenol material, Cavit, and the fast setting zinc oxide-eugenol cements Kwikseal, No-Mix, Dentin, Tem-Pac, and Kalsogen. The results showed that those materials which sealed poorly at room temperature also performed poorly after thermocycling. Of the materials which did effect a seal at room temperature only Cavit, Kwikseal and amalgam did not permit dye penetration after thermocycling.

The sealing ability of Cavit placed over medicated (camphorated parachlorophenol) and nonmedicated cotton pellets was assessed by Webber et al. (1978). They concluded that dye penetration with 10% aqueous methylene blue was solely dependent on the thickness of Cavit and that a thickness of 3.5 mm was necessary to prevent leakage. No thermocycling was done in this study. They also found that the

penetration of the dye within the material itself was the same as that at the tooth-filling interface and felt that the leakage potential of Cavit was due to its hydroscopic properties.

When investigating the effect of temperature on the sealing properties of Cavit and Cavit G, Oppenheimer and Rosenberg (1979), found that a depth of 2mm of material was adequate to prevent leakage of 2% methylene blue in all but one tooth restored with Cavit G and two teeth restored with Cavit.

In 1982 Tamse et al., compared the sealing ability of IRM, Kalsinol, (both zinc oxide eugenol based), Cavit, Cavit G, and Cavidentin, (calcium sulfate based with no eugenol). They used a thermocycling method similar to that of Parris et al (1960), with 1% methylene blue and 0.5% eosin as dyes. They found that eosin showed significantly less penetration than the methylene blue, highlighting the effect that dye selection may have on results. Cavidentin showed a significantly superior seal to IRM, Kalsinol and Cavit. The seal was better than that obtained with Cavit G but not significantly so.

Composite resin both chemical and light cured used as a temporary seal has been compared with Cavit, zinc phosphate cement, and zinc oxide eugenol cement (ZOE), the more customary temporary sealing agents used in endodontics (Chohayeb and Bassiouny, 1985). Access cavities and the root canal system were prepared as they would be during routine endodontic therapy with 2.5% NaOCl used as an irrigant. A space of 2.5mm was left for the material to be placed over a dry cotton pellet. After a 24 hour maturation period in tap water the specimens were cycled between 4 °C methylene blue and 58 °C water 40 times. Cavit was found to provide the best seal and where dye penetration was seen it was slight and had a low intensity of staining. The composite resins showed greater sealing ability than the ZOE and zinc phosphate cement although 60% showed some degree of marginal leakage. The light cured composite showed a slightly better result than the chemical cure composite. It should be noted that no acid etch of the enamel was performed in this study, and there was no mention as to whether an unfilled resin was used.

Fayyad and Shortall (1987), investigated dye penetration along the enamel and cementum walls of class II MOD composite restorations where bond enhancing agents had been used. They showed that although capable of reducing leakage where enamel was present at the margin, dentine bonding agents perform poorly adjacent to dentine or cementum. The only test group resulting in less marginal leakage on the cementum side as compared to the enamel side was that using Ketac-bond a GIC lining cement. It must be noted that no group showed margins free from leakage. The best results obtained were those of the Gluma system and Clearfil New Bond against an enamel margin.

In a recent study Robbins and Cooley (1988), looked at the microleakage of Ketac-Silver, a glass cermet cement in tunnel preparations and class V preparations and showed leakage in both groups. The only persistantly leak proof group was that in class V cavities with the placement of a varnish covering. However the protective film would wear off eventually in the oral environment. From this study it appears that the bond of the material to dentine and enamel did not prevent the penetration of dye.

Recently a new temporary endodontic material, TERM (Temporary Endodontic

Restorative Material), has been introduced. The material is a light cured resin based system and is claimed to "provide a durable, tight seal for endodontic access cavities. The material offers protection against microleakage between visits and placement of the final restoration" (Caulk, Dentsply. Form no. 570990).

Teplitsky and Meimaris (1988), compared the performance of TERM and Cavit in a dye leakage study using 10% methylene blue. Each temporary material was placed to a depth of 4mm over a cotton pellet in access cavities in anterior, premolar and molar teeth. The groups of teeth were placed into water for 30 minutes and then placed into the dye. Half the specimens were stored at room temperature and the rest thermocycled with 28 one hour exposures from 4 °C to 60 °C over a 7 day period. After sectioning, the cotton pellets were inspected for dye and margins investigated for degree of dye penetration. It was found that Cavit had an effective seal in 91.7% of cases and that thermal variation did not adversely affect its ability to maintain a seal. TERM only maintained an effective seal in 33.3% of cases and thermal variation caused more leakage. In this study the seal using Cavit was significantly better statistically than that using TERM.

Other work comparing the sealability of TERM and Cavit has shown the opposite results to those above. McDonald et al. (1988), in an <u>in vivo</u> study comparing TERM, Hard TERM and Cavit, randomly filled access cavities with the test materials and left them for a minimum of 30 days (average 46). Staining with caries disclosing solution showed Hard TERM to have the least penetration at the margins after removal of the material with high speed burs without water spray. It also showed the least marginal staining and discolouration before application of the dye. TERM was the next best and

Cavit the least effective seal.

Ludlow and Hermsen (1988), also investigated the sealing ability of TERM comparing it with Cavit and Cavit-G in 57 extracted human molars. The teeth underwent 600 thermal cycles over 24 hours before staining with 50% silver nitrate solution for three hours, and exposure to a photographic flood light to blacken the remaining silver nitrate. TERM was found to have significantly less penetration and the depth of penetration was seen more as a function of the absorption into the material rather than the lack of marginal seal.

Several materials with different coefficients of thermal expansion were examined in class V cavities using 0.5% basic fuschin dye after thermocycling, (Bullard et al., 1988). The results showed a direct correlation of increased marginal leakage with an increase in the coefficient of thermal expansion of the material. Glass ionomer was closest in coefficient of thermal expansion to that of tooth at  $14 \times 10^{-6}$ /C and had the least leakage. See table 2.1 for a summary of results.

#### TABLE 2.1 Effect of Coefficient of Thermal Expansion on

#### Microleakage

	Coefficient of	Value for degree	
MATERIAL	thermal expansion	of dye penetration	
Sevriton	80	6.00	
Silux	57	5.63	
Fynal	35	4.85	
Occlusin	24	4.20	
Cluster	22	2.05	
Ketac-Fil	14	1.55	

Results from Bullard et al. (1988).

Coefficient of thermal expansion as  $10^{-6}$ /°C.

Microleakage grading:

1. No detectable mocroleakage.

2. Penetration of dye into enamel only.

3. penetration of dye into dentine.

4. Partial involvement of the axial wall of the cavity.

5. Total involvement of the axial wall.

6. penetration of dye into dentinal tubules.

7. penetration of dye into the pulp chamber.
#### 2.4.2 Bacterial penetration studies

Seltzer (1955), used colour producing organisms to assess the penetration of these organisms under the margins of acrylic and amalgam restorations. The tests carried out at both constant body temperature and after thermocycling found that organisms only penetrated the margins after the restorations had undergone a temperature change.

In addition to the dye penetration studies that they had carried out Parris et al. (1964), used motile bacteria to investigate further the problem of marginal leakage. The bacteria at approximately 500 micrometres in diameter were in the order of 250 times larger than the analine blue dye they had used in the previous study (1960). The organisms used were Sarcina lutea which can remain viable at 60 °C for a minimum of 60 minutes and Serratia marcescens which can remain viable at 4 °C for a minimum of 60 minutes. The 130 teeth tested were cycled ten times through broths of the organisms at 4 °C and 60 °C. Of the materials tested only five showed no bacterial leakage - Kalsogen (Ash), Zinc oxide-eugenol, Kwikseal, Cavit and amalgam.

In 1977 Krakow et al. conducted an <u>in vivo</u> study of 7 temporary filling materials, each material tested in the same anterior tooth in subjects which had had satisfactory endodontic treatment completed on the tooth. Cavit, Caviton and zinc oxide-eugenol were shown to perform similarly with over 80% of teeth showing no or only minor leakage. The zinc phosphate cements showed no leakage in more than two thirds of teeth. This differs from the findings of Parris et al. (1964), who did observe leakage around zinc phosphate fillings.

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An <u>in vivo</u> study in monkeys to assess the microleakage of Cavit was conducted by Lamers et al. (1980). The anterior teeth of 45 rhesus monkeys were used, access cavities were sealed with 2mm of Cavit-W and the teeth were tested for leakage at 2, 7, and 42 days. The results indicated that after 2 days 23% of teeth were positive for bacterial infection, after 7 days 37%, increasing to 69% at 42 days. The investigators did not feel that the increase in bacteria found after longer periods was due to the higher chance of bacteria being detected after multiplying, as the 2 day group should then show a percentage higher than the 23% found. At no time period was the 2mm thick Cavit 100% impervious to microleakage. With increased periods of exposure there was a greater chance of microbial penetration. This would indicate that the maximum thickness should be used and that inter appointment periods should be no longer than a week unless a double seal is used.

#### **2.4.2.1 Effect of softening of materials by drugs**

Keller et al. (1981), and Blaney et al. (1981), examined the microleakage of temporary restorations over cotton pellets soaked in saline or camphorated monochlorophenol (CMCP) to determine whether the softening of the materials by CMCP might have an effect on microleakage. It had been observed (Olmstead et al., 1977) that IRM was softened more than Cavit or zinc phosphate cement. Proteus vulgaris was used as it is one of the most penetrating and motile organisms available.

Samples were taken at 1, 2, 3, 4, 5, 13 and 21 days in the Keller et al. study. Due to the very small sample size in the study only trends could be seen from their results; however the CMCP softened IRM compared favourably for sealability with the IRM set

against saline. All models sealed with Cavit and IRM against saline showed leakage within 13 days.

Blaney et al. (1981), conducted a study similar to that of Keller et al. (1981), with a larger sample size. The results showed that IRM set against CMCP provided a significantly better seal than other samples tested. IRM set against saline had a seal no better than Cavit against CMCP except at the two day level. Both materials performed significantly better when set against CMCP and it was felt that the residual effect of the medicament was responsible for this. At three weeks the majority of samples had been penetrated. Cavit set against saline had 100% penetration at two weeks. IRM performed better than Cavit in all cases except when set against saline at the one week interval.

Both these studies indicate that IRM and Cavit do not provide leak-proof seals and that the time between appointments should be kept to a minimum where possible.

#### 2.4.3 Radioactive isotope studies

Marosky et al. (1977), investigated the microleakage of six materials as temporary seals using <sup>45</sup>Ca as the tracer and testing at three and ten days after placement of the materials. Materials tested were a zinc phosphate cement, Fleck's cement, zinc oxide-eugenol cement (U.S.P.), IRM, Duralon (a polycarboxylate cement), Cavit and Temp-Seal. Three hundred and sixty teeth were stained and then had autoradiographs taken of washed and dried longitudinal sections. Half the teeth were thermally cycled 500 times between 10 °C and 50 °C for 30 seconds in each bath. The results showed that in general the leakage increased as the fillings aged and after thermal cycling. The ranking

of the materials from best to worst was Temp-Seal (removed from the market due to lead content), Cavit, zinc oxide-eugenol cement, zinc phosphate cement, IRM and Duralon. Only Temp-Seal and Cavit failed to have specimens with severe leakage under any of the test conditions. The authors felt that manipulative factors had an influence on the results with Temp-Seal and Cavit, both being single paste materials not requiring mixing.

A study aimed at the immediate and early sealing properties of Cavit using a radioisotope solution of aqueous sodium sulfate and autoradiography was conducted by Todd and Harrison in 1979. Total marginal penetration was seen in 15 of 18 test restorations with maturation periods of 15 minutes or less. Also marginal penetration was greater than surface penetration with only 3 of 22 sections showing total isotope penetration through the Cavit mass as compared to 16 of 22 with total marginal penetration.

Welsh and Hembree (1985), investigated the leakage of <sup>45</sup>Ca around class V abrasion type lesions with one margin in dentine and the other in enamel. Several dentine bonding agents and one glass ionomer cement were studied at one week, three months and six months. The glass ionomer alone showed no or slight penetration at all time intervals. All the bonding agents showed marked leakage for all time intervals.

A study investigating the sealing ability of Cavit and zinc oxide-eugenol in recently placed amalgam (Tytin) and composite resin (Consise) was conducted by Orahood et al. (1986), using <sup>45</sup>Ca. Most previous studies had only investigated cavities prepared in sound teeth. Clinically it is more likely to find the need to prepare the

access cavity in a tooth with an existing restoration which may form part or all of the cavity margin. Thermocycling was carried out for 2500 cycles of 30 second duration. Although no significant difference was seen between the two materials zinc oxide-eugenol provided the better seal against resin and Cavit the better seal against amalgam. There was significantly greater microleakage when the access was prepared through amalgam as compared to composite resin.

#### 2.4.4 Radiodiffusion studies

Four temporary filling materials were assessed from their ability to prevent leakage of radiosodium, (Friedman et al., 1986). The materials tested were of two types - zinc oxide-eugenol based (IRM, ZOE) and calcium sulphate based ( Cavit-G, Cavidentin). Sampling for <sup>22</sup>Na was carried out at 4,8,and 24 hours, then once daily during the first week and every 2-3 days during the following three weeks. All four materials demonstrated leakage at a constant rate with the difference in leakage between the materials of the same class being insignificant. After day three, the difference in leakage of the four materials was significant, with the zinc oxide-eugenol materials performing better than the calcium sulphate based materials. Day six to seventeen showed the differences to be highly significant, the differences remained significant to day nineteen and thereafter were no longer significant. Cavit-G leakage was significantly higher than that of IRM from day three to twenty three, Cavidentin leakage was higher than that of IRM and ZOE but not significantly so. The authors stated that "the amount of leakage demonstrated by IRM and ZOE fillings (the better seals in this study) after 23 days indicates that even these materials should not be recommended for endodontic use for longer than 3 weeks."

Powis et al. (1988), carried out a long term monitoring of the microleakage of glass ionomer, polycarboxylate, silicate and zinc phosphate cements. They used a radiochemical diffusion model with <sup>14</sup>C as a tracer and tested the specimens at one day, one week, six weeks and fifty two weeks. They found that the adhesive cements and particularly the glass ionomer cements provided an excellent seal (often no leakage detected) in nearly all cases which was maintained for the full 52 weeks. The silicate cements showed erratic behaviour but had a far worse seal while the zinc phosphate cements showed marked leakage at all time periods.

#### 2.4.5 Other studies

Weber et al. (1978), in their study on the sealing quality of Cavit also carried out a small scanning electron microscope (SEM) evaluation of the adaption of the cavit to the access cavity wall. They found good adaption in most of the peripheral areas but did find crevices ranging from  $37\mu m$  to  $63\mu m$  in some regions. The possibility that these crevices were as the result of artefact during specimen preparation was not investigated.

The effect of pretreatment of dentine with 5% sodium hypochlorite (NaOCl) or a 0.2% EDTA containing commercial cavity cleaner, (Tublicid), on the marginal adaption <u>in vivo</u> of a glass iomoner cement, a glass cermet and three dentine adhesives was investigated using the SEM (Dijken van and Horstedt, 1987). The fillings placed in 52 class V cavities had a one month intra-oral period and were then extracted and sectioned bucco-lingually for SEM investigation (replication technique used). Neither of the pretreatment regimens prevented contraction gaps forming with the resinous adhesive systems. Pretreatment with sodium hypochlorite resulted in gap free margins with the glass ionomer and glass cermet cements and only small gaps between 0.5 and 4 micrometres wide were seen with these materials when Tublicid was used. The glass ionomer and glass cermet cements with 5% NaOCl pretreatment showed the best adaption.

Anderson et al. (1987), used a fluid filtration technique to compare the sealing ability of 4 mm thick IRM, Cavit and TERM in access cavities. Measurements were taken at one hour, 24 hours, and seven days. The restorations were then thermocycled 60 times between 4 °C and 56 °C, with two minutes at each temperature, and microleakage measured again. Cavit and TERM provided leakproof seals during the time interval studied. IRM showed significantly greater leakage at seven days and after thermocycling. Additional tests carried out for eight weeks on the TERM fillings showed no deterioration of the seal.

#### 2.5 PHYSICAL PROPERTIES OF TEMPORARY ENDODONTIC MATERIALS

#### 2.5.1 Introduction

Although the integrity of the seal of temporary endodontic sealing materials can be seen as the most important aspect during the various treatment stages of endodontic therapy, the importance of the other physical properties of the material being used must not be overlooked. It is the ability of the material to withstand the oral environment that will result in the mantenance of a seal for the required time period. This certainly becomes increasingly important where long term dessings are being utilized.

Properties that will determine whether a material is suitable include;

- 1. setting time,
- 2. ease of manipulation,
- 3. dimensional stability,
- 4. strength,
- 5. abrasion resistance,
- 6. solubility and disintergration, and
- 7. adhesion to tooth and restorative materials.

The majority of work published on the various physical properties of the dental cements is related to their use as cementation materials and thus at powder liquid ratios that differ from those used when the material is prepared for use as a temporary restorative.

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#### 2.5.2 Setting time

The International Organization for Standardization (ISO) has set out the minimum setting times for dental cements of filling consistencies in the standards ISO 1566-1978 (E), ISO 3106-1974 (E), ISO 3824-1984 (E) and ISO 4104-1984 (E). These times are listed below.

#### TABLE 2.2 Recommended ISO setting times

	Maximum setting
MATERIAL	time at 37°C
Zinc phosphate	8 m 30 s
Zinc oxide-eugenol	7 m
Silicophosphate	5 m
Polycarboxylate	5 m

The setting time of cements is affected by several factors both during manufacture of the material and at the time of mixing in the dental surgery. Generally the major factors under the control of the dentist, are the temperature and the powder liquid ratio. In looking at resin reinforced zinc oxide eugenol materials Civan et at (1972), found setting times to vary between materials from a low of 2 minutes with ZOE, (B&T) at a powder liquid ratio of 4.1 gm/ml to a high of 11 minutes with IRM at a powder liquid ratio of 1.5 gm/ml.

Several polycarboxylate materials of luting consistency with a powder liquid ratio of 1.5:1 by weight were tested by Powers et al. (1974). Generally the materials showed setting times of 6 to 7 minutes at 37 °C and 100% relative humidity.

Glass ionomer-cements at luting consistencies have been shown to have setting times in the order of 6.5 minutes for Ketac-Cem to 8.5 minutes for Fuji 1 and Chembond (Mc Comb et al., 1984).

Fast setting glass-ionomer cements were developed after the discovery by Wilson and Crisp (1972), that the incorporation of optically active d-tartaric acid increased working time and increased the setting rate of glass-ionomer. Most commercially available filling consistency glass-ionomer cements form a hard cement within 10 minutes from the start of mixing (Wall, 1986).

The majority of composite resin based materials used today are light activated usually with a minimum exposure time of 20 seconds to set the material. As a result of this technology setting time is of little concern with these materials.

On the other hand Cavit has a far greater setting time than the materials mentioned above. It requires exposure to the water present in the oral cavity to initiate

setting and takes in the order of an hour to develop a hard set.

#### 2.5.3 Manipulation

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The ease of manipulation of a material may have an effect on its performance in the longer term. Failure may result from incorrect handling. The more complex the manipulative requirements are to place a material, the more likely it is that they may not be correctly carried out.

Marosky et al. (1977), in looking at the marginal leakage of temporary sealing materials felt that the superior seal seen with Temp-seal and Cavit over the zinc oxide eugenol, zinc phosphate, IRM and Duralon cement tested was due to the fact that these materials were premixed and did not introduce the variables of mixing. They also noted that "IRM seemed grainy and without much body after mixing; therefore, it did not pack into the opening as well as the others did."

The need to hand mix many cements leads to a great variation in the powderliquid ratios which can effect the strength and wear resistance of a material. For this reason IRM has been marketed in encapsulated form. However even this provides mixes of varying consistencies depending on the trituration. The manufacturers advise that the first five capsules be used to establish the best mixing time for any particular amalgamator.

The recently developed light cured composite material, TERM, has exellent manipulative characteristics, being easy to dispense place and cure (Teplinsky and

Meimaris, 1988). It is injected directly into the cavity with a single use compule and then light activated.

The manipulative requirements of the glass-ionomer cements are, in comparison to the other materials discussed, most exacting, as there is a critical water balance. Dehydration of the material results in loss of water needed for cement formation, causing fissuring and cracking. Excess water before hardening will result in the washing out of the cement thus weakening the surface and making it susceptible to erosion. It is therefore necessary to protect the surface of the cement with a varnish or light cured bonding agent (McLean, 1988). It is obvious that a great deal of care is required when placing these materials.

#### 2.5.4 Dimensional stability

The dimensional stability of a material will greatly influence its ability to maintain a marginal seal over an extended time period. The coefficient of thermal expansion of the material is an important factor in this regard. Other important factors are contraction or expansion on setting, or exposure to water in the oral environment, which may lead to the disruption of the seal.

Gilles et al (1975), conducted a study on the dimensional stability on temporary restoratives, in which they looked at Cavit, IRM and an unmodified zinc oxide-eugenol cement. They examined the percentage linear change with temperature change on dry heating. They found that all materials displayed shrinkage on heating in a dry chamber at 37 °C for two hours as a result of water loss. In comparing the materials the dimensional changes seen with Cavit on cycling between 22 °C and 55 °C were small as compared with the other materials. They point out that dimensional stability is dependent on hydration equilibrium as well as other thermodynamic characteristics, and that the ability of a material to absorb water can lead to marked expansion in the oral cavity.

The manufacturers claim that the one week water sorption (wt%) for TERM is 2.3-2.9 % and for Cavit is 15.3 % indicating that TERM is a far more stable material. This is to be expected as the setting reaction for TERM is not water dependent as is that of Cavit.

Widerman et al. (1971), in looking at the physical properties of cavit showed the percentage weight increase with time caused by water sorption at 72 hours to be 5.37  $\pm$  0.43 % for Cavit and 1.13  $\pm$  0.43 % for zinc oxide-eugenol. They also showed Cavit to expand linearly 14.2  $\pm$  0.09 % as compared to 8.40  $\pm$  0.8 % for zinc oxide-eugenol after 10 days in distilled water at 37 °C. These findings concluded that water sorption by a material will lead to expansion. More importantly it was shown that a three hour water sorption figure of 9.6 % for Cavit was actually 17.99 % when it was discovered that 8.39 % of its weight was lost in that time period as a result of solubility and disintegration.

It is apparent from the studies of Widerman et al. (1971), and Gilles et al. (1975), that water sorption is a major determinant in expansion of materials and that Cavit undergoes a large expansion during its hygroscopic setting. This gross expansion of Cavit on setting is seen as the factor responsible for producing the relatively good sealing properties seen with this material.

A factor which may lead to gap formation is the polymerization shrinkage that occurs on curing. This is particularly relevant to composite resin systems. In an attempt to overcome this problem, the acid etch technique (Kopel, 1971) was developed as well as the use of dentine bonding agents, and these have been shown to reduce leakage (Ebright et al., 1985; Hansen, 1986; McComb et al., 1986). This is however not supported by all investigators (Davila et al., 1986).

Composite resins have also been shown to undergo hygroscopic expansion after placement which can assist in eliminating gap formation. However the length of time required to achieve compensation for the polymerization shrinkage may be up to seven days. The degree of dimensional change with water sorption decreases as the filler content increases (Hirasawa et al., 1983).

Wilson and McLean (1988), have found that glass-ionomer cements undergo only minor dehydration shrinkage if placed in a humid environment.

#### 2.5.5 Strength

Zinc phosphate cement at a powder liquid ratio of that for luting has a compressive strength in the order of 80-110 MPa and a tensile strength of 5-7 MPa at 24 hours (Smith, 1983; Branco and Hegdahl, 1983). These workers have also shown polycarboxylate to have a compressive strength of 55-85 MPa and a tensile strength of 8-12 MPa. Polycarboxylate cements are less brittle and more resiliant than zinc

phosphate and glass-ionomer cements.

Glass-ionomer cement (luting consistency) has a compressive strength of 90-140 MPa and a tensile strength of 6-8 MPa after 24 hours with the strength increasing over this period (Smith, 1983, and McComb et al., 1984).

Drummond et al. (1988), have shown that after two years of aging in distilled water zinc phosphate cement showed a small, but not statistically significant, loss of compressive strength, while polycarboxylate and glass-ionomer cements showed an increase in compressive strength.

In a study of three resin reinforced zinc oxide-eugenol cements at powder liquid ratios ranging from 1.5:1 to 6.8:1 it was found that while B&T, a ZOE cement, showed little change in compressive strength (39-45 MPa), or tensile strength (4-4.7 Mpa), over this range, IRM and Fynal showed increases in strength, with IRM's compressive strength increasing from 42 to 89 Mpa and its tensile strength from 3.4 to 8.2 MPa. Fynal's compressive strength increased from 57 to 76 MPa and its tensile strength from 6 to 9.6 MPa (Civjan et al., 1972).

The strength of the reinforced zinc oxide-eugenol materials is much greater than that of unmodified zinc oxide-eugenol which has a maximum compressive strength value of 40 MPa (Smith, 1983). EBA cements have a compressive strength value of 50-70 MPa and a tensile strength value of 6-7 MPa (Jendersen et al., 1969; Smith, 1983).

Cavit has a compressive strength of approximately 14 Mpa according to

Widerman et al. (1971), however the TERM product profile claims this to only be 2 Mpa.

TERM is claimed to have a compressive strength of 40-48 MPa and a tensile strength of 5-7 Mpa. Other composite materials have higher compressive strengths ranging to 300 Mpa (Powers et al., 1983) and tensile strengths in the order of 45 Mpa (Phillips, 1982) depending on filler type and percentage.

#### 2.5.6 Solubility, disintergration and wear resistance

A great deal of emphasis has been placed on the solubility and disintegration of dental cements especially in the case of luting type materials. The standards rely on solubility and disintegration in distilled water as an indication of the ability of a material to withstand breakdown. This can be extremely misleading. Dissolution in weak organic acids and alkalis can be many times greater than that seen with water, and different materials will react differently in these solutions.

In the oral cavity lactic and pyruvic acids are found and would affect the dissolution of material (Coleman and Kirk, 1965).

As a result of this awareness many of the more recent studies have used weak acid solutions to assess solubility, <u>in vitro</u>.

Many investigators have used weight loss to compare differences between materials. However the great variation in rate and magnitude of water sorption and loss between materials leads to inaccuracies in test results (Walls et al., 1985), thus making interpretation of meaningful comparisons difficult.

Another method using residual weight also presents inaccuracies as this only measures residual non volatile agents, and in the case of a material containing eugenol, which is volatile, may greatly underestimate the actual degree of disintegration of the material (Widerman et al., 1971).

From the above remarks it can be seen that a great deal of <u>in vitro</u> data on solubility must be viewed with caution in reference to the clinical situation.

Wear resistance and solubility and disintegration are closely related factors. Norman et al. (1969), have shown an interesting relationship between combined <u>in vitro</u> wear and solubility data, with <u>in vivo</u> wear. Results show a close linear relationship between the combined <u>in vitro</u> data and the <u>in vivo</u> material. Certainly it could be expected that a material which has a high solubility in the oral environment would show a low wear resistance in this same environment.

When assessing wear tests one can look at ranking materials, which is most commonly done with a two body wear test. Alternatively the mechanism of wear, is often determined using a single pass test (Craig, 1979). The majority of tests which have been carried out on dental cements are of the two body wear test form.

Craig (1979), has shown that the rate of wear is dependent on the hardness of the abrasive used, and that composite materials with glass fillers wear almost twice as fast as those containing quartz fillers. Thus the nature of the filler is a major determining

factor, in addition to percentage of filler in determining composite wear. Another factor affecting wear is the surface treatment of the filler particles which may enhance the retention in the resin matrix.

Listed in the tables below are data from several sources relating to solubility and disintegration.

# TABLE 2.3 Weight loss of dental materials from distilled

water solubility studies

AUTHOR	P/boxylate	ZOE	ZnP0	Cavit	EBA TERM
Wederman					% of residual
et al 71		0.34%		9.73%	weight 7 days
TERM form				(e)	
570990	7 day			15.3%	0.3 - 0.4%
				%	of residual
Smith 83	0.1-0.6%	1.5%	0.04-3.3	% v	veight 23 hrs
Coleman		A 0.08%	A &c	B are b	oth experimental
Kirk 65		B 0.2	0.2%	fortifie	ed materials
Norman	1 day	0.39	0.26	residu	1al weight
et al 69	5 day	0.72	1.00	(m	g/sqcm)
Jendresen		A 1-2 ui	nmodified	I ZOE	
et al 69	all	ex	kperimen	tal EBA	B 20-30
	5 day	C 0-1 re	einforced	ZOE	
(mg/sqcm)		D 0-1 re	einforced	surface	e modified ZOE

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The following table shows data from studies using weak acid solutions

<b>TABLE 2.4</b>	Weight loss of	dental	materials	from	weak	acid	solubility	studies
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AUTHOR P	/boxylate	ZOE	ZnPO	Cavit	GIC	EBA	TERM
Norman	1 day	0.83	1.65	dilute	acetic ad	eid pH	4
et al 69	5 day	2.08	8.91	(mg/se	lcm)		
Jendresen	5 day	A 10-12	unmod	ified ZO	E		
et al 69	10 <sup>-4</sup> M ac	etic	experin	nental E	EBA I	B 40 +	
	acid	C 9-11	feinford	ced ZOE			
(mg/sqcm)	pH 4	D 8-10	reinfor	ced sur	face mod	lified Z	ZOE
Mc Comb	% of resi	dual weig	ht 0.1 M	f lactic	acid pH	4 24	hr
et al 84	0.16%		0.59%		0.56-1.43	3%	
Walls	221.67		49.49		16.08-60	).26	
et al 85	um dep	th loss af	ter 6 h	rs conti	nuous		
	erosion	cycling in	n 0.1 M	lactic a	acid pH 4	1	

The study conducted by Walls et al. (1985), is interesting because the method involved cycling of specimens for periods of 90 seconds in alternate baths of distilled water or the eroding solution. The gentle washing action resulting from repeated immersions removed any loosely bound debris thus exposing fresh cement for the following cycle. Unfortunately with this study, as with most others, comparison between it and other studies is not possible as a result of the different methodologies. From all the data listed above it is only possible to suggest rankings of materials. Even ranking of materials must be viewed with some reservations when the complications and inaccuracies possible, as noted above, are considered. Information as to their possible behaviour intraorally cannot be interpreted from these results.

There have also been some <u>in vivo</u> solubility and disintegration studies carried out (Richter and Ueno, 1975; Mitchem and Gronas, 1978, 1981; Osborne et al., 1978; Pluim et al., 1984; Ibbetsson et al., 1985) and when this and the previous data are considered a ranking of these materials can be given.

MOST RESISTANT	- glass-ionomer
	- silicophosphate
	- zinc phosphate
	- polycarboxylate
LEAST RESISTANT	- zinc oxide-eugenol

The majority of the above solubility data relates to luting type materials, rather than filling type materials, which generally have a higher powder liquid ratio.

Wear or abrasion data have also been collected for <u>in vitro</u> and <u>in vivo</u> studies for various cements. Powers et al. (1983), in looking at composite resins, compared <u>in vivo</u> and <u>in vitro</u> data for the same materials and found correlation values of less than 0.8. These results led to the suggestion that <u>in vitro</u> results could be used in screening tests for new materials.

Whether this would be true for cement type materials has yet to be determined, but as mentioned earlier, Norman et al. (1969), have presented data showing a close linear relationship between the combined <u>in vitro</u> wear and solubility data, and <u>in vivo</u> data.

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Swartz et al. (1963), determined that a mechanical toothbrushing machine, of the type used for testing the abrasive qualities of dentifrices, was suitable for testing the abrasion resistance of cements because it permitted a wide choice of abrasives and minimized the danger of clogging which may occur with abrasive discs. The results of their study, using weight loss to determine the degree of wear, found that silicate and zinc phosphate cements to be far more resistant to wear than zinc oxide-eugenol materials.

Norman et al. (1969), concurred with the results of Swartz et al. (1963), with an <u>in vitro</u> one hour brushing on a toothbrushing machine, wear data with percentage weight loss for the materials tested being 0.6% for silicate, 3.0% for zinc phosphate and 20.8% for zinc oxide-eugenol cements. Only the zinc oxide-eugenol material was of a high powder liquid ratio (6.25:1). <u>in vivo</u> data over a 30 day period, with these materials in windows on the lingual aspect of a partial denture, ranked the materials in the same order as that seen in the <u>in vitro</u> study.

Jendresen et al. (1969), placed zinc oxide materials in a similar toothbrushing machine to that used by Swartz et al. (1963), and Norman et al. (1969), and found that reinforced zinc oxide materials showed greater wear resistance than a conventional and an experimental EBA zinc oxide material.

Composite resin materials have a variable degree of wear with some recently developed hybrid materials having wear rates approximating that of amalgam (Dogon and Van Leeuwen, 1985; Lambrechts et al., 1985). Certainly the wear resistance of these materials is greater than that of the dental cements.

TERM is a resin based material; however its filler particle is also a resin material rather than the glass type fillers seen in restorative composite resins. As a result its wear resistance cannot be considered as that of a composite resin. The manufacturers warn that if long periods of service are required that IRM, a resin reinforced zinc oxide-eugenol should be used. This would indicate that TERM has a relatively poor wear resistance.

#### 2.5.7 Adhesion to tooth

Adhesion is the force that is produced when molecules are attracted and which makes two substances attach to one another when they are brought into contact. This force is the result of Van Der Waals forces and chemical bond formation (Phillips, 1982).

Of the materials available for use to seal access cavities, only the polycarboxylate and glass ionomer cements adhere to tooth structure chemically. The precise mode of adhesion is unclear but it has been suggested that phosphate groups of the apatite crystal in tooth structure are displaced by carboxyl groups from the polyacid, thus providing a chemical link between the two molecules. Calcium ion loss from the tooth maintaining electrical neutrality and thus the stability of the bond (Walls et al., 1986; McLean, 1988).

The bond strengths of GICs and polycarboxylate materials have been measured by various investigators and are listed below.

# TABLE 2.5 Tensile bond strengths of polycarboxylate and

	Polycarboxylate		Glass-i	onomer
AUTHOR	Enamel	Dentine	Enamel	Dentine
Tsuburaya				
et al 84	2.3	1.5		
Thornton				
et al 86	•		1.2-2.3	0.5-2.0
Jemt				
et al 86	1.7-2.8		1.2-2.4	

glass-ionomer cement to tooth structure

Resin based materials may be bonded to the tooth via micromechanical interlocking of resin tags into irregularities in enamel produced by acid etching, and to dentine with the aid of bonding agents. Different dentine bonding agents have been shown to have variable bond strengths to dentine with values ranging from 0.2 to 3.7

MPa (Chan et al., 1985). Another complication with some dentine bonding agents is that the bonds may undergo hydrolysis on exposure to moisture and fail (Causton, 1984).

The surface treatment of dentine and enamel with various chemical agents does affect the bond strength attained with poly-electrolyte cements and it has been shown that polyacrylic acid and tannic acid were the most effective conditioners (Wilson and Prosser, 1984).

It must be remembered that the endodontic access cavity may have been exposed to chelating agents such as EDTA. Prolonged exposure to such agents may adversely affect the bonding of materials as many of these rely on the inorganic phase of enamel and dentine to produce the bond. It has been shown that Tublicid (0.2% EDTA) treatment of cavities for 60 seconds and 20 second acid etching of enamel margins resulted in gap formation with Glass-ionomer materials (4 micrometres maximum) and dentine bonding agents (12 micrometres maximum). Pretreatment with 5% NaOCl for 30 seconds resulted in gap free cavities with the glass-ionomer materials, but gaps of a similar magnitude as that seen with Tublicid for the dentine bonding agents (van Dijken and Hörstedt, 1987).

#### 2.6 ANTIMICROBIAL PROPERTIES OF TEMPORARY SEALING MATERIALS

The aim of a temporary sealing material is primarily to prevent the ingress of organisms. A material with antibacterial properties would be advantageous. This will of course not affect its ability to prevent the ingress of small toxins or substrate particles which may support the growth of existing organisms in the root canal.

In a study by Krakow et al. (1977), zinc oxide-eugenol was shown to have an antibacterial effect on saliva inoculated blood agar plates, showing an inhibitory zone of 1 to 2 cm. Cavit although also bacteriocidal had a smaller inhibitory zone of less than 1 cm.

Schwartzman et al. (1980), who investigated a number of materials using five different organisms on blood agar plates, confirmed the bacteriocidal nature of zinc oxide-eugenol. Second in efficacy was silicophosphate cement followed by zinc phosphate cement. Polycarboxylate cement and a composite resin material showed no inhibitory effect on most of the organisms tested.

The efficacy of the zinc oxide-eugenol is attributed to the free eugenol present in the material.

Tobias et al. (1985), used a similar method to that of Schwartzman et al (1980), to assess several materials including glass-ionomer materials. They found that all the materials tested showed some antibacterial activity when freshly mixed, with the ranking from best to worst being, zinc oxide-eugenol, silicate, glass-ionomer cement and zinc phosphate cement. Significantly they showed that after seven days the only material to still show significant antibacterial properties was zinc oxide-eugenol.

In an attempt to improve the antibacterial properties of these materials, workers have combined chlorhexidine with the cements and found that this does substantially increase these properties. They have also shown that the physical properties of the materials are not significantly affected with bacteriocidal doses (Schwartzman and Caputo, 1982; Jedrychowski et al. 1983).

#### 2.7 LITERATURE SUMMARY

Endodontic treatment aims essentially at the elimination of the microbiological agents and the subsequent placement of inert materials to seal the canal system from reentry of microorganisms.

During the course of endodontic therapy it is important that contamination of the root canal system is prevented. A temporary restorative material is used to seal the root canal system from the oral environment and prevent the ingress of organisms, their toxins or material which may enhance the growth of existing bacteria within the root canal.

Although the ability of a temporary filling material to provide a leak proof seal is of primary importance, other factors, such as the durability of the material over customary appointment intervals, will ultimately affect the seal developed and hence the efficacy of these materials.

It is obvious from reviewing the literature that there is a variety of methods available to assess the seal produced by a restorative materials. However many of these methods can only be used <u>in vitro</u> and extrapolation of results to the clinical setting is difficult and may be misleading.

It appears that many zinc oxide-eugenol based materials perform poorly as a seal in preventing the ingress of the various agents used to demonstrate leakage when compared with Cavit and TERM. While the performance of all materials deteriorates after thermocycling, the zinc oxide-eugenol materials are those most adversly affected.

There have been conflicting reports on the sealing ability of the adhesive dental cements, polycarboxylate and glass-ionomer. This may relate to the fact that these materials are extremely sensitive to moisture contamination and dehydration.

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Bacterial penetration and radioisotope diffusion studies have shown that, regardless of the material used, leakage will worsten with time, and that no material tested can maintain an impervious seal for greater than three weeks. For this reason it has been recommended that treatment intervals should be kept to a minimum (Keller et al., 1981; Blaney et al., 1981; Friedman et al., 1986). Lamers et al. (1980), after assessing results from an <u>in vivo</u> study of bacterial leakage recommended that intervals between appointments should be no longer than a week and that if a greater interval was anticipated a double seal is used in an attempt to improve the longevity of the seal.

It is also important to recognize that most studies have only looked at leakage in virgin teeth. Clinically it is more likely to find the need to prepare the access cavity in a tooth with an existing restoration which may form part or all of the cavity margin. In the only study reviewed involving the preparation of cavities in amalgam and composite resin Orahood et al. (1986), found that there was significantly greater microleakage when the access was prepared through amalgam as compared to resin.

The physical properties of most of the commonly used materials are relatively poor and incapable of withstanding long intraoral periods.

It would appear that Cavit and TERM are suitable sealing agents in routine cases with short inter appointment periods and that the newer glass-ionomer materials may also prove to be satisfactory.

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AIM

The aim of this research is to assess the suitability of several dental materials, and some epoxy resin based industrial materials, for use as a long term temporary endodontic coronal sealing agent.

Dental materials not customarily used restoratively, and the industrial materials, were to be assessed <u>in vitro</u>, in conjunction with dental materials already used extensively as access cavity seals, to allow some indication of possible clinical performance of materials as compared to data collected on those materials already in use.

It was not the aim of this study to develop a new material but rather to indicate which of a series of materials might show promise for further investigation and modification.

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This was a preliminary study of several classes of material and toxicity studies were not carried out at this stage, but would need to be addressed subsequently on any material undergoing further investigation. Tests performed were those which were thought to have greatest relevance to the possible clinical performance of a material, namely;

setting time, microleakage during thermal cycling, dimensional stability, rate of wear <u>in vitro</u>, effect of medicaments, and <u>in vivo</u> wear.

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These were to be carried out sequentially as a sieve with a material being discarded from further investigation if it failed to perform adequatelly.

#### 4

## **MATERIALS AND METHODS**

#### 4.1 MATERIALS

Both dental and industrial materials have been investigated in this study and are as follows (see appendix I for manufacturer details of all materials and equipment used).

#### 4.1.1 Dental test materials

1. Cavit, a single paste material based on zinc oxide, calcium sulphate, and zinc sulphate.

2. Genesis, a light cured polyether, urethane dimethacrylate resin impression material (the syringe low viscosity material was used).

3. Impregum F, a polyether impression material (heavy body).

4. IRM CAPS (Intermediate Restorative Material Capsules), an encapsulated resin reinforced zinc oxide-eugenol temporary filling material. A trituration time of 10 seconds was used.

5. Ketac-Bond Caps, an encapsulated quick set glass-ionomer lining material. A trituration time of 10 seconds was used.

6. Permadyne, a newer polyether impression material (heavy body).

7. Ramitec, a polyether based bite registration material.

8. TERM (Temporary Endodontic Restorative Material), a light cured resin based (urethane dimethacrylate) temporary endodontic restorative material.

#### 4.1.2 Industrial test materials

1. Araldite 5 Minute epoxy resin adhesive, combined with 400 grit aluminium oxide in a powder: liquid ratio of 2:1 to provide a material with a greater viscosity than the resin alone.

2. Loctite E-POX-E Ribbon Putty, an epoxy putty with an initial cure of two hours and a final cure of 12 hours which can set under water.

3. Loctite 10 Minute E-POX-E Ribbon Putty, a similar material to the loctite epoxy putty with an initial cure of 10 minutes and final cure of two hours.

### 4.1.3 Material preparation

All trituration was carried out on an Ultramat high velocity amalgamator.

Light cured materials were cured with a Translux CL light source.

Where indicated Slipicone silicone releasing agent was used as a lubricant.

Test proceedures were carried out in the sequence listed below, each test acting as a sieve for those that followed, with testing on a material being discontinued if it failed to pass the preceeding test.

#### 4.2.1 Setting time

The setting time of each material was initially determined by observation of the material at room temperature. In this way the consistency and behaviour of the material could be assessed for suitability and the material discarded from further study if necessary.

Following this each material where appropriate was tested using an oscillating cement rheometer as described for Resin-based Dental Filling Materials (BS 5119, 1975). The apparatus was prepared as directed in the standard and water at 37 °C was circulated through the test plates.

Materials such as the elastomers and epoxy resins which demonstrated a marked degree of flexibility on setting were tested on the rheometer specifically designed for testing elastomeric materials as described in British Standard 4269 Part 1. (1968), Specification for Dental Elastic Impression Materials, Part 1. Elastomeric impression materials.

The materials were mixed according to the manufacturers' instructions and placed

between the test plates which had been lubricated with a silicone releasing agent. The variations in amplitude were monitored on a Heath Schlumberger Strip Chart Recorder System (EU-205B), to determine the setting time. Setting time was calculated from the start of mixing or in the case of Cavit from the time of immersion in water. A modification of the lower plate to provide a well around it allowed the setting of Cavit to be recorded under water, all other materials were tested dry.

4.2.2 Microleakage

#### 4.2.2.1 Specimen preparation

The sealability of materials was tested in teeth, amalgam composite resin and a glass cermet cement. Premolars which had been extracted for orthodontic reasons were stored in a tooth preserving solution (chlorhexidine gluconate 0.05% with cetrimide 0.5% in aqueous solution). Access cavities were cut using a fine tapered fissure tungsten carbide bur at high speed with air-water spray cooling. Root canals were prepared with EDTAC solution used as an irrigant to size 25 file and 1% NaOCl for size 30 and above files. The teeth were finally irrigated with the EDTAC solution. Following canal preparation retrograde cavities were prepared in each tooth which was then stored in distilled water for 24 hours at room temperature.

Amalgam, composite resin and glass cermet cement specimens were prepared in single plastic blocks (Lego). Amalgam was mixed according to the manufacurers instructions packed into the blocks and allowed to set. Herculite XR light cured composite resin was placed into the blocks in bulk and cured for 20 s from each end of
the block. The glass cermet cement was injected into the plastic blocks and allowed to set for 10 minutes; these specimens were then stored in distilled water for two weeks before manipulation to avoid dehydration effects. Cavities were cut in the amalgam and glass cermet cement specimens with a cross cut fissure tungsten carbide bur at high speed with air-water spray to a depth of 6 mm and these specimens then rinsed and stored in distilled water. A fissure diamond bur was used to cut 4 mm deep cavities in the resin specimen which were then stored in distilled water (these specimens were shallowed due to the limited availability of material).

Ten teeth and ten amalgam specimens were randomly selected for each test material. Specimens were removed from storage in distilled water and air dried. A cotton pellet was placed into the depth of the cavity leaving a minimum of 3 mm for placement of the material. Retrograde preparations in tooth specimens were filled with E-POX-E putty to prevent leakage via the apical foramen. Test materials were mixed according to manufactures' directions and then placed into the prepared cavities with a flat plastic instrument. Exceptions to this were TERM, Genesis and Ketac-Bond which were syringed into place with their respective delivery systems and the polyether materials which were syringed into place with a plastic syringe (Application-Syringe). Light cured materials (TERM and Genesis) were cured for 40 s from the occlusal surface. Ketac-Bond was allowed to set for 4 minutes prior to placement in distilled water and no protective varnish was used.

After the test materials had been placed, specimens were allowed to mature for 24 hours in distilled water at 37 °C. Tooth specimens were then air dried and coated with two layers of nail varnish to within 1 mm of the cavity margins, the second layer

being placed after the first had dried (approximately 15 minutes). Once the second coat had dried specimens were thermocycled.

### 4.2.2.2 Thermocycling

Each specimen was thermocycled for a period of 100 cycles between 4 °C and 60 °C  $\pm$  2 °C with immersion periods of 25 seconds at each temperature. The machine used consisted of two water baths, one kept at 4 °C with ice and the other at 60 °C with a heater unit. Suspended in each bath was a container of 1% methylene blue dye. A basket containing the specimens, which was situated on the end of an oscillating arm, was immersed in one of the methylene blue containers for 25 s and then transferred to the other methylene blue container. This is shown in figure 4.1. A period of 5 s was required to transfer the basket. After completion of 100 cycles specimens were rinsed in water to remove excess dye and sectioned.

Only those materials which demonstrated a satisfactory seal in the tooth specimens were further tested in the composite resin and glass cermet cement preparations. The sequence for their placement, maturation and thermocycling was as for the tooth and amalgam specimens.

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THERMOCYCLING



 FIGURE 4.1
 A. The prepared specimens and thermocycing apparatus are shown

 diagramatically (MB - methylene blue dye).
 B. A photograph of the

 apparatus in use.
 100 cycles with 25 second immersion times were made

 for each sample group.

Specimens were sectioned through the mid section of the access cavity using a diamond disk (EM Cutting Disk) at 12,000 rpm with water spray.

# 4.2.2.4 Assessment of leakage

The degree of leakage of dye was assessed under a stereomicroscope and classified according to the following criteria:

0 = No marginal leakage evident,

1 = Marginal leakage evident but less than 1 mm of penetration,

2 = Marginal leakage with greater than 1 mm of penetration but no complete penetration,

3 = Total leakage.

Penetration of dye through the material itself was also noted.

Stainless steel moulds approximately 4 mm in internal diameter and 8 mm high were used for specimen preparation.

The moulds were lubricated with a thin film of silicone releasing agent (Slipicone) and then filled with the test materials in the same manner as the access cavities for the microleakage study. Five specimens were prepared for each material. Light cured materials were cured for 40 seconds from each end of the mould. The surfaces of the set materials were sanded flush with the surface of the moulds and an initial measurement made with a vernier calliper. Specimens were then placed into distilled water and stored at 37 °C. Measurements of each specimen after gentle air drying were again taken at one and fourteen day intervals.

Circular plastic disks 35 mm in diameter and 4 mm thick were prepared with six 5.5 mm diameter holes to accept test materials.

A single material was placed into the six holes in a disk and stored in distilled water at 37 °C for 24 hours. The surface of the material was then sanded flush with the surface of the disk and placed into the wear machine.

The wear machine consisted of a bristle brush with a load of 98 grams in conjunction with Zircate prophylaxis paste as an abrasive provided the wearing action (figure 4.2). The disk had an outer and inner flanges placed over the edges of the test material to provide a datum line for measurement purposes; the outer flange also acting as a container for the Zircate paste (figure 4.3). The specimen disk was rotated under the brush at 300 rpm for a three hour period. During this time regular additions of fresh Zircate paste were made to ensure a constant wearing action.

The surface of the disk was replicated with a polysiloxane (Permagum) which was sectioned for measurement; this was carried out under a stereomicroscope against a calibrated objective slide (figure 5.22).





FIGURE 4.2 The wear machine and its components are shown diagramatically A and photographically B. Test material placed in a plastic disk was worn at 300 rpm for a period of three hours by a brush loaded with 98g and with the regular addition of fresh Zircate paste as an abrasive.

B

A





FIGURE 4.3The wear disk with holes to accept test materials with the brass inner<br/>flange and plastic outer flange are shown seperately A, and assembled B.<br/>The overlaping of the test material by the flanges to produce a datum for<br/>measurement can be seen in B.

#### 4.2.5 Effect of Ledermix paste and Pulpdent paste on set materials

A 4 mm thick plastic sheet had 5.5 mm diameter holes prepared in it to accept test materials. These materials were placed into the holes in the same manner used for the access cavities for the microleakage study. Samples were allowed to mature for 24 hours and then sanded flat.

Double sided tape was used to adhere fibrous washers to the plastic and thus create a well around each sample (figure 4.4a). Six samples of each material were used with Ledermix paste and Pulpdent paste each being placed into two wells and two samples left as controls. A glass slab was placed over the washers sealing the wells and thus preventing the pastes from dehydrating (figure 4.4b). This complex was then placed at 37 °C and 100% relative humidity for seven days.

After the test period the washers were removed and the pastes washed from the material surface with running water. The surfaces were then air dried.

A Shore A durometer (Zwick) was then used to determine the hardness of each surface.

Where any change in surface colouration was seen in the test material the specimen was removed from the plastic mould and sectioned to determine the depth of penetration.





FIGURE 4.4 A fibrous washer was adhered with double sided tape to the plastic sheet, to provide a well around test materials, to allow for placement of medicaments, A. A diagram illustrating the assembly of the apparutus is

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shown, B.

B

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A palatal acrylic partial denture was used to test <u>in vivo</u> wear. Six wells were created to accept test material. Three wells were placed adjacent to the occlusal surfaces of the posterior teeth on each side (figure 4.5). The denture was to be worn for a period of one month.

Cavit, TERM and Permadyne were each placed into two wells and allowed to mature in water for 24 hours (TERM light cured for 20 seconds). The surfaces of the materials were then made as smooth as possible and flush with the denture surface with sand paper. The surface of the denture was then replicated with a polysiloxane impression material (Permagum).

Unfortunately after three weeks of wear little observable wear had occured with any material and the test was discontinued.

The worn surface was to be replicated and compared to the initial replica made.



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FIGURE 4.5 An acrylic plate was made with sites prepared for test materials adjacent to occlusal surfaces of the posterior teeth, A. This was worn by the investigator and is seen shortly after insertion, B.

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Where appropriate, data was analyzed statistically using either the  $\underline{t}$  test, one way ANOVA, or chi square test. These tests were all carried out using a Microstat statistical package.

The Student Newman-Keul multiple comparison procedure was used to show which means differed for significant ANOVA procedures.

Where 2x2 chi square tests resulted in any cell containing a value less than 5 a two tailed Fishers exact probability test was carried out using the BMDP 4F programme.

# **RESULTS**

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# 5.1 Setting time

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Setting time data is listed in table 5.1. Setting times were calculated according to the appropriate standard for the two different testing machines used, to the nearest five seconds. A graphic representation of setting times is shown in figure 5.1.

Examples of the chart recordings from the oscillating rheometers are shown in figure 5.2.

Term and Genesis, light cured materials and E-POX-E ribbon putty with a set time of two hours were not assessed.

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## TABLE 5.1

# SETTING TIME

SAMPLE						
MATERIAL	1	2	3	4	5	MEAN <u>+</u> STD
CAVIT	1500	1260	1320	1800	1680	1512 + 206
IRM	260	435	450	225	380	350 + 92
KETAC-BOND	260	275	270	265	275	269 <u>+</u> 6
	Oscillatin	ng Rheome	er data			
RAMITEC	170	185	180	150	160	169 + 13
IMPREGUM F	250	240	270	270	265	259 + 12
PERMADYNE	385	270	250	240	265	282 ± 53
E-POX-E 10	360	330	570	560	500	464 + 101
ARALDITE	290	325	335	340	340	326 ± 21
	Rheomet	er for elas	tomeric ma	terials dat	a	

# SETTING TIME EXPRESSED IN SECONDS

All materials set dry except Cavit which was set under water. Setting times were calculated to the nearest 5 seconds.



# FIGURE 5.1 Setting times of test materials. Where the oscillating cement rheometer was used an \* is placed next to the material name. All other materials were tested on the oscillating elastomer rheometer.



FIGURE 5.2Examples of readings from the oscillating cement rheometer are shown<br/>at A Ketac-Bond, B Cavit, and C IRM CAPS (half actual size). Examples<br/>of readings from the oscillating elasomeric rheometer are shown at, D 10<br/>Minute E-POX-E putty, E Araldite 5 Minute in a powder liquid ratio of<br/>2:1 with aluminium oxide, and F Permadyne. Arrows indicate the point<br/>at which the materials set.

The results are shown in table form and graphically for the test materials in each four sites, tooth, amalgam, composite resin, and Ketac-Silver (tables 5.2 - 5.5, figures 5.3 - 5.6).

# 5.2.1 Microleakage in tooth

To assess microleakage in tooth 110 premolar teeth were prepared with 10 teeth selected randomly for each material to be tested; seven of these teeth were discarded from investigation due to tooth fracture and dye penetration along these fractures. Examples of specimens after sectioning are presented in figures (figures 5.7 -5.10).

The significantly poorer performance of IRM and Ketac-Bond can easily be seen from figures 5.3 and 5.8.

# 5.2.2 Microleakage in amalgam

To assess microleakage in amalgam 110 amalgam blocks were prepared for investigation with 10 allocated for each test material. No blocks were discarded. Examples of sectioned specimens are shown in figures (5.11 - 5.14).

Chi square analysis of the seal of test materials in amalgam showed a significant difference between the test materials at the 1% level (for statistical purposes leakage clasifications of 0 and 1 were taken as representing a satisfactory seal and clasifications

of 2 and 3 as representing an unsatisfactory seal). Fishers exact probability tests showed the sealability of TERM to be significantly different to that of Cavit, the polyethers and epoxy resin based materials at the 1% level but not significantly different to that of Ketac-Bond. The seal of Ketac-Bond in amalgam was not significantly different to that of any of the other test materials (figure 5.4).

Investigations on IRM and Ketac-Bond were discontinued due to poor performance in tooth and amalgam microleakage studies.

### 5.2.3 Microleakage in composite resin

To assess microleakage in composite resin 45 composite resin blocks were prepared for investigation with 5 allocated for each test material. No blocks were discarded. All materials tested sealed satisfactorily in composite resin (figure 5.5). Examples of sectioned specimens are shown in figures 5.15 and 5.16.

# 5.2.4 Microleakage in Ketac-Silver

To assess microleakage in Ketac-Silver 45 Ketac silver blocks were prepared for investigation with 5 allocated for each test material. one block was discarded as a result of shattering during sectioning. Examples of sectioned specimens are shown in figures (5.17 and 5.18).

Chi square analysis showed no significant difference in the seal of materials tested in Ketac silver (figure 5.6).

# 5.2.5 General observations

In general the manipulation of the test materials in the method previously described (4.2.2.1) was easy but the Araldite placement was an exception to this, being difficult to insert without incorporation of air bubbles (figure 5.14).

For a few specimens Leakage of dye between the amalgam and plastic block lead to some difficulties during sectioning where dye was smeared over the cut surface and into the cotton wool pledget of some specimens. Fortunately this only affected one half of the specimen and the other was available for assessment. This problem was over come for composite resin and Ketac-Silver specimens by removing the plastic blocks from the samples before sectioning.

# DEGREE OF LEAKAGE IN TEETH

		SATISF	ACTORY SEAL	UNSATISFACTORY SEAL	
MATERIAL	Ν	0	1	2	3
CAVIT	10		10		
IRM	10				10
TERM	0	3	5		
KETAC-BOND	10		1	4	5
GENESIS	9	8	1		
RAMITEC	9	9			¥
IMPREGUM F	10	10			
PERMADYNE	10	10			
E-POX-E 10	9	8	1		
E-POX-E	9	9			
ARALDITE	9	9			

N = Total number of specimens

0 = No leakage

1 = Leakage of < 1 mm

2 = Leakage of > 1 mm but not complete

3 = Complete leakage





as being unsatisfactory.

#### UNSATISFACTORY SEAL SATISFACTORY SEAL 3 0 ı 2 Ν MATERIAL 10 CAVIT 10 9 1 10 IRM 5 3 2 10 TERM 3 1 KETAC-BOND 10 6 7 3 10 GENESIS 10 RAMITEC 10 IMPREGUM F 10 10 2 8 10 PERMADYNE 10 10 E-POX-E 10 9 1 10 Е-РОХ-Е 9 L ARALDITE 10

## DEGREE OF LEAKAGE IN AMALGAM

N = Total number of specimens

0 = No leakage

1 = Leakage of < 1 mm

2 =Leakage of > 1 mm but not complete

3 = Complete leakage

#### MATERIAL





as being unsatisfactory.

# TABLE 5.4

# DEGREE OF LEAKAGE IN COMPOSITE RESIN

		SATISFA	SATISFACTORY SEAL		UNSATISFACTORY SEAL	
MATERIAL	N	0	L	2	3	
CAVIT	5		5			
TERM	5	5				
GENESIS	5	5				
RAMITEC	5	5				
IMPREGUM F	5	5				
PERMADYNE	5	5	51			
E-POX-E 10	5	5				
E-POX-E	5	5				
ARALDITE	5	5				

N = Total number of specimens

0 = No leakage

l = Leakage of < 1 mm

2 = Leakage of > 1 mm but not complete

3 = Complete leakage





# TABLE 5.5

# DEGREE OF LEAKAGE IN KETAC-SILVER

		SATISFA	CTORY SEAL	UNSATISFACTORY SEAL		
MATERIAL	N	0	L	2	3	
CAVIT	5		5			
TERM	5	5				
GENESIS	5	5				
PERMADYNE	5	5				
Е-РОХ-Е 10	5	3	2			
E-POX-E	4	2	Ł	Î		
ARALDITE	5	5				

N = Total number of specimens

0 = No leakage

1 = Leakage of < 1 mm

2 = Leakage of > 1 mm but not complete

3 = Complete leakage









FIGURE 5.7 A. Sectioned tooth specimen of Cavit after thermocycling. The level of dye penetration seen was consistent for all cavit specimens regardless of substrate. B. TERM specimens showed some leakage of dye through the material but to less than 1 mm for all tooth specimens. (10 X magnification).





FIGURE 5.8 A. All IRM seals placed in tooth showed complete leakage. B. 90% of Ketac-Bond seals placed in tooth showed either complete leakage as seen here or leakage greater than 2mm marginally which was considered unsatisfactory. (16 X magnigication).





FIGURE 5.9 A. All 10 Minute E-POX-E putty seals placed in tooth sealed completely (10 X magnification). B. The Araldite aluminium oxide mixture also showed 100% complete sealing in tooth (16 X magnification).



FIGURE 5.10 All polyether materials showed 100% complete sealing of tooth specimens. A Genesis. B Permadyne. (10 X magnification).



FIGURE 5.11 All IRM seals placed in amalgam A, showed complete leakage (10 X magnification). 80% of TERM seals in amalgam showed either complete leakage or leakage greater than 2mm marginally (16 X magnification).



FIGURE 5.12 80% of Ketac-Bond seals placed in amalgam were satisfactory showing no leakage A, or leakage of less than 1mm marginally. The other specimens showed unsatisfactory seals B. (16 X magnification).





FIGURE 5.13 Both polyether based materials (Permadyne A) and epoxy resin based materials (2 hour setting E-POX-E putty B) showed no leakage for all amalgam specimens. (10 X magnification).



FIGURE 5.14 Although most specimens of the Araldite aluminium oxide mixture were properly placed A (10 X magnification), the material was hard to manipulate, and incorperation of air bubbles difficult to avoid B (16 X magnification).



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FIGURE 5.15 A. The dye penetration shown in this specimen of Cavit in composite resin was consistent for all specimens (10 X magnification). B. All TERM specimens in composite resin showed no marginal leakage (16 X magnification).



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FIGURE 5.16 Specimens of Genesis A (16 X magnification), and 10 Minute E-POX-E putty B (10 X magnification), in composite resin cavities are shown. All materials tested in composite resin sealed satisfactorily.



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FIGURE 5.17 Specimens of Cavit A, and TERM B, in Ketac-Silver cavities are shown. No leakage is seen for TERM and leakage to less than 1mm marginally is seen for Cavit. (10 X magnification).



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**FIGURE 5.18** Specimens of Genesis A, and 10 Minute E-POX-E putty B, in Ketac-Silver are shown. (10 X magnification).

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Measurements taken initially at 24 hours and 14 days are presented in table 5.6. The percentage linear expansion demonstrated by each material at 24 hours and 14 days is shown in figure 5.19.

Statistical analysis using the t test showed significant differences between the 24 hour and 14 day linear expansions for TERM, Ramitec, Impregum F, Permadyne, 10 Minute E-POX-E putty and the Araldite mixture at the 1% level. There was no significant difference between the two time intervals for Cavit, Genesis and the 2 hour setting E-POX-E putty (figure 5.19).

A significant difference was also shown between 10 Minute E-POX-E putty and the 2 hour setting E-POX-E putty at both 24 hour and 14 days at the 5% level.

A one way ANOVA test was carried out on the polyether based materials for both the 24 hour and 14 day test periods and showed a significant difference at the 1% level for the two time periods. Further analysis using the Student Newman-Keul test showed that the only polyether materials not to be significantly different from each other at the 5% level were Genesis and Ramitec for the 24 hour test period.
### TABLE 5.6

MATERIAL AND	
TIME PERIOD	MEAN % EXPANSION
CAVIT 24 HOUR	9.0 + 0.8
CAVIT 14 DAY	$10.0 \pm 0.0$
TERM 24 HOUR	0.2 + 0.1
TERM 14 DAY	$0.8 \pm 0.2$
GENESIS 24 HOUR	1.4 + 0.2
GENESIS 14 DAY	$1.5 \pm 0.3$
RAMITEC 24 HOUR	1.5 <u>+</u> 0.3
RAMITEC 14 DAY	$4.0 \pm 0.8$
IMPREGUM F 24 HOUR	$0.4 \pm 0.3$
IMPREGUM F 14 DAY	$2.6 \pm 0.1$
PERMADYNE 24 HOUR	0.0 <u>+</u> 0.3
PERMADYNE 14 DAY	$1.3 \pm 0.2$
E-POX-E 10 24 HOUR	0.1 <u>+</u> 0.1
E-POX-E 10 14 DAY	$1.4 \pm 0.1$
E-POX-E 24 HOUR	$0.5 \pm 0.4$
E-POX-E 14 DAY	$0.9 \pm 0.5$
ARALDITE 24 HOUR	0.6 <u>+</u> 0.2
ARALDITE 14 DAY	2.7 + 0.1

#### PERCENTAGE LINEAR EXPANSION

Linear expansion of materials in distilled water at 37°C.





Measurements of depth of wear are presented in table 5.7. This is expressed graphically in figure 5.20.

The plastic disks carrying the materials were shown to wear at the rate of the material present. The wear was also shown to be bristle dependant as indicated by grooving (figures 5.21).

Examples of the sectioned replicas of the worn surfaces that were used for measurement are shown in figure 5.22.

A one way ANOVA showed a significant difference between the wear depths seen for the test materials at the 1% level (figure 5.20). Further analysis using the Student Newman-Keul test showing where the differences lie is shown in figure 5.23.

### TABLE 5.7

## WEAR DEPTH

MATERIAL	WEAR DEPTH IN	MICRO	METRES	
CAVIT	449	<u>+</u>	32	
TERM	193	+	31	
GENESIS	142	+	52	
RAMITEC	150	<u>+</u>	63	
IMPREGUM F	153	+	52	
PERMADYNE	74	+	22	
E-POX-E 10	166	<u>+</u>	57	
Е-РОХ-Е	335	<u>+</u>	117	
ARALDITE	85	+	20	

Test conditions: 3 hours at 300 rpm with Zircate paste and a brush load of 98 grammes.









FIGURE 5.21 Different rates of wear can be seen between Cavit A, the material showing greatest wear, and Permadyne B, the material showing least wear. Grooving can be seen within the wear path. (10 X magnification).



FIGURE 5.22 Examples of sectioned Permagum replicas of the worn surfaces are shown for, A Cavit, B TERM, and C Permadyne. These were measured under a stereomicroscope against a calibrated objective slide. (16 X magnification).

Least wear PERMADYNE ARALDITE GENESIS IMPREGUM F RAMITEC E-POX-E 10 TERM E-POX-E CAVIT

Greatest wear

FIGURE 5.23 The Student Newman-Kuel showed that the materials joined by a line did not have significantly different depths of wear at the 5% level.

Visual inspection of all materials at the end of the test period demonstrated surface staining on all surfaces exposed to Ledermix paste (figure 5.24). Pulpdent paste did not stain surfaces .

Sectioning of the Ledermix paste samples showed minimal penetration of this staining effect, indicating that this was essentially a surface effect only (figure 5.25).

Hardness tests on material surfaces showed no significant differences between the control and test specimens except for Permadyne (table 5.8). However this result is not consistent for that seen with the other polyether materials and should be regarded with suspicion.



FIGURE 5.24 A. Cavit control without staining. B. Cavit specimen exposed to Ledermix paste showing staining. C. TERM control without staining. D. TERM specimen exposed to Ledermix paste showing staining. Samples of test materials were exposed to the medicament for 7 days at 37 °C and 100% relative humidity. (10 X magnification).





FIGURE 5.25 Sectioned Genesis A, and Permadyne B, specimens which had been exposed to Ledermix paste shows that the staining by the medicament was essentially a surface effect. (10 X magnification).

### TABLE 5.8

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# **EFFECT OF MEDICAMENTS ON HARDNESS**

MATERIAL	CONTROL	LEDERMIX PASTE	PULPDENT PASTE
CAVIT	97.5 + 0.5	97.5 + 0.5	97.7 <u>+</u> 0.5
TERM	99.0 + 0.0	99.0 <u>+</u> 0.0	99.0 + 0.0
GENESIS	64.8 + 5.0	63.8 <u>+</u> 1.7	67.5 + 2.0
IMPREGUM F	98.2 <u>+</u> 0.4	99.0 <u>+</u> 0.0	99.0 <u>+</u> 0.0
PERMADYNE *	56.8 <u>+</u> 0.8	53.2 <u>+</u> 0.8	52.7 <u>+</u> 0.8
E-POX-E 10	$59.3 \pm 3.1$	57.7 <u>+</u> 1.0	61.7 <u>+</u> 2.3
ARALDITE	94.0 + 0.6	93.5 <u>+</u> 0.5	93.7 <u>+</u> 0.5

Means values are shown. Measurements were taken with a

Shore A durometer. \* indicates the only material to show a

significant difference between means at the 5% level.

## 5.6 <u>In vivo</u> wear

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The test materials failed to show any wear capable of measurement over a period of three weeks. It was felt that the site of placement of materials in the test appliance was most likely responsible for this as they failed to be involved in the masticatory cycle. As a result of this the test was discontiued.

# DISCUSSION

#### 6.1 Selection of test materials

The dubious performance of traditionally used temporary restorative materials particularly in the long term is well documented in the literature where investigations using several different methodologies have been made (Marosky et al., 1977; Todd and Harrison, 1979; Lamers et al., 1980; Keller et al., 1981; Blaney et al., 1981; Tamse et al., 1982; Chohayeb and Bassiouny, 1985; Friedman et al., 1986; Teplinsky and Meimaris, 1988). In all these studies the only substrate in which materials were tested was tooth, and rarely was there exposure of the cavity surfaces to currently used endodontic irrigants.

The only reported study investigating the seal of temporary materials in restorative materials was that of Orahood et al. (1986), however the small number of samples precluded meaningful conclusions from being made.

Materials investigated included dental materials traditionally used as temporary access cavity seals, polyether based dental materials and epoxy resin based industrial materials.

Marosky et al. (1977), found that many of the powder-liquid materials that they tested had an inferior seal as compared with Cavit and Temp-Seal both single paste

materials. They felt that the easier manipulation of the single paste material was a factor in their better performance. For this reason encapsulated IRM and Ketac-bond were used to minimize the effects of manipulation and to assist in the standardization of powder:liquid ratios.

The polyether based dental materials were also chosen for investigation because of their ability to closely adapt to surfaces, their fast setting and the fact that exposure to moisture will result in expansion of the material and this could enhance any seal that might be achieved.

Epoxy resins are a class of materials which are seldom used in dentistry with AH26 root canal sealer being an exception. The epoxy resin putties investigated (E-POX-E putty and 10 Minute E-POX-E putty) have a consistency which allows for easy manipulation and were claimed by the manufacturer to set under water, adhere well to metals and have low toxicity. Araldite 5 Minute epoxy adhesive had 400 grit aluminium oxide added to provide an epoxy material with a hard wearing filler component and a putty consistency.

### 6.2 Setting time

The time that a material may take to set will influence its selection by an operator, as in the clinical situation it is inconvenient and often impractical to use materials with long setting times. However a material such as Cavit may have a long setting time but not require full setting before the patient is dismissed because of adequate stiffness and apparent adequate sealability in the unset form. As the 2 hour

setting E-POX-E putty was found to be stiff after mixing it was assessed beyond this point.

Cavit has been shown to seal well once set (Tamse et al., 1982; Chohayeb and Bassiouny, 1985; Teplinsky and Meimaris, 1988), however Todd and Harrison (1979), have however shown that the early sealability of Cavit is poor. The early leakage probably occurs during hygroscopic setting expansion where it is taking up water and expanding to provide a closer adaption to the cavity walls.

Standards using oscillating rheometers and giving chart recordings were used to assess setting times for the materials tested because they gave some indication from the shape of the graphs drawn of the setting characteristics of each material. The oscillating elastomer rheometer was used for the epoxy resin and polyether based materials because they had too much residual flexibility for the oscillating cement rheometer used with the traditional restorative materials.

Ketac-Bond showed a substantial setting period with little amplitude change, where the material would still be relatively easy to manipulate, followed by a rapid decrease in amplitude to the set point showing that once the material starts to set that the reaction is then rapid to the final set (figure 5.1a). In contrast Cavit (figures 5.1b) showed a continuous and relatively constant decrease in amplitude showing a continual setting reaction from its initial immersion in water. IRM showed a setting pattern similar to that of Cavit over a shorter time frame (figure 5.1c).

The polyether materials (figure 5.1f), and the Araldite aluminium oxide mixture

(figure 5.1e), appeared to show a rapid set at the end of a period of little amplitude change on the graph. The 10 Minute setting E-POX-E putty showed a more gradual and prolonged setting reaction (figure 5.1d).

All materials tested other than Cavit set in less than 10 minutes. IRM and 10 minute setting E-POX-E putty showed a large range of setting times and it is most likely that this was due to manipulative variables as evidenced by the observation that the incorporation of the powder in the IRM capsules after trituration was often incomplete. The difficult in obtaining equal proportions of both components for the 10 Minute E-POX-E putty was another manipulative variable encountered.

The information leaflet accompanying Cavit does not indicate the setting time of the material other than to say that it hardens quickly. In this study Cavit was found to take approximately 25 minutes from it initial immersion in water. In light of its proven poor initial seal this may detract from its value as a temporary sealing material.

#### 6.3 Microleakage

Teeth which had been stored in a tooth preserving solution for various periods of time were used in this study. It would have been advantageous if freshly extracted teeth could have been used as some specimens contained cracks which allowed dye leakage. These specimen had to be discarded from the study. Although the teeth were endodontically prepared to try and approximate the clinical situation the preserving solution may have affected the results. Amalgam, light cured composite resin and Ketac-Silver were also chosen as substrates in which to test for leakage as these materials are often present in teeth undergoing endodontic therapy. To date only one study has examined the sealing of materials in a material other than tooth (Orahood et al., 1986). This demonstrated a better seal when the access cavity was prepared in composite resin as compared to amalgam.

Methylene blue dye was selected to disclose any leakage as it had been shown to penetrate further than isotope tracers (Matloff et al., 1982). Tamse et al. (1982), also showed methylene blue to have significantly greater penetration than eosin. A pilot study showed methylene blue to penetrate further than eosin. It also was easier to detect.

Crim and Garcia Godoy (1987), have shown that results from a thermocycling study on composite resins were not significantly different when samples were cycled 100 times as compared to 1500 times. They also demonstrated that there was no difference between immediately cycling the specimens or allowing them to mature in distilled water for 24 hours. For this reason 100 cycles was chosen for this study and specimens were allowed to mature in distilled water for 24 hours as setting times varied between 3 minutes and 2 hours.

Baths of methylene blue dye were used to stain specimens during the cycling process as it was felt that the greatest amount of leakage would be seen at that stage. In many studies samples are thermocycled and then stained.

The results from this section of the study demonstrated that IRM proved a poor

seal in tooth and amalgam specimens ( 100% leakage). This is in agreement with other IRM leakage studies, with and without thermocycling, (Marosky et al., 1977; Tamse et al., 1982; Anderson et al., 1987). IRM has been shown to perform significantly better than Cavit-G to the twenty third day of testing in a radiodiffusion study using <sup>22</sup>Na (Friedman et al., 1986).

The poor performance of Ketac-Bond in tooth specimens where 90% of seals proved to be unsatisfactory, may be due to the exposure of the tooth surface to EDTA for extended periods of time during endodontic preparation. It could be postulated that a high percentage of the calcium on the cavity surface which is necessary for bond formation was chelated from the surface by the EDTA. This coupled with a setting contraction may explain this poorer than expected result. As 70% of seals in amalgam were satisfactory (leakage to less than 1mm) the problem is not wholly related to contraction. The poor results for Ketac-Bond in this study might not apply to the light cured glass-ionomer cements such as Vitrabond which have been recently released.

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As a result of these poor performances IRM and Ketac-Bond were excluded from further investigation.

TERM showed good sealing properties in all substrates other than amalgam where only 20% of seals were satisfactory. This was significantly poorer than all materials other than Ketac-Bond tested in amalgam.

All polyether materials showed an excellent seal in all substrates. This is probably due to the close adaption to the cavity wall and expansion on contact with water, as demonstrated in the dimensional stability test. The next stage of testing for this material would to determine the effect of mechanical cycling on this apparently excellent seal.

The epoxy resin based materials also performed very well and may provide a new class of materials suitable for the temporary restoration of cavities although the question of biocompatability still needs to be addressed. The manufacturers of the E-POX-E putties claim that they adhere to almost any surface, particularly metal surfaces, set under water and have low toxicity. They certainly form a relatively stiff putty on mixing and are relatively easy to manipulate.

The diglycidyl ether of bisphenol A (DGEBA), the basic component of all epoxy resins is relatively non toxic systemically and has no known carcinogenicity. It is the curing agents that are the problem often causing skin irritation. Once the epoxy resin is set it is generally inert and non toxic (Lee and Neville, 1967).

#### 6.4. Dimensional stability

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This study confirmed the large expansion seen with Cavit on setting and demonstrated that after 24 hours any further increase in expansion was not significant. The main part of this initial expansion probably takes place during the setting reaction which was found to take approximately 25 minutes. Widerman et al. (1971), showed a percentage weight increase of 9.6% in Cavit specimens due to water sorption at 3 hours. They indicated that water sorption was the major factor responsible for the setting expansion.

The dimensional stability study showed an interesting difference in the 24 hour and 14 day results for Genesis when compared to the other polyether based materials. Genesis was the only one of these materials to show no significant continual expansion beyond the 24 hour period. It is postulated that the light cured nature of the material and its urethane dimethacrylate component may be responsible for this. Certainly Genesis showed a 24 hour expansion which was significantly greater than that seen for the other polyether materials except Ramitec.

The epoxy resin based materials, except the 2 hour E-POX-E putty, expanded significantly beyond the 24 hour test interval, indicating that they continued to absorb water over this period.

#### 6.5 Wear study

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The objective of the <u>in vitro</u> wear test was to rank test materials in order of wear rate. Cavit and the 2 hour E-POX-E putty showed significantly greater wear than the other materials tested, with Cavit showing the greatest. Permadyne showed the least wear, but this was only significantly different to that of TERM, 2 Hour E-POX-E putty and Cavit at the 5% level when analyzed using the Student Newman-Kuel test.

This wear test provided a relatively harsh environment with Zircate prophylaxis paste as an abrasive and a wear period of 3 hours and 300 rpm, never the less long term clinical use of a material will expose it to a high level of wear particularly on the occlusal surface of molars.

Unfortunately the design of the <u>in vivo</u> wear apparatus did not allow as much wear as was expected. As Cavit showed very little or no detectable wear at 3 weeks the test was discontinued. The wear pattern for Cavit seen clinically is variable with some patients showing very little wear while in others the material may be almost completely removed within a short time.

It had been hoped that a comparison of <u>in vivo</u> and <u>in vitro</u> wear with the materials tested could give some indication of the possible clinical performance of the tested materials. It would also have allowed us to gain some idea of what level of wear seen on the experimental apparatus would be satisfactory clinically.

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Work carried out by Powers et al. (1983), on composite resin showed a high correlation value between <u>in vitro</u> and <u>in vivo</u> wear. He suggested that <u>in vitro</u> wear results could be used as screening tests for new materials.

The brushing machine used in the <u>in vitro</u> wear study produced wear by passing a loaded brush in an abrasive paste over the surface of the test material. This aims at providing a similar action to the abrasion of food across the surface of a material during chewing.

The use of a brushing machine was chosen in preference to an abrasive disk (Powers et al. 1983), as it minimized the danger of clogging which may occur with an abrasive disk.

The brushing machine model has been used by many investigators (Swartz et al.,

1963; Norman et al., 1969; Jendresen et al., 1969).

The wear pattern seen on the disks showed that the plastic wore at the same rate as the test material placed in it, indicating that the effect of the wear of the disk itself on the results would be minimal. A new brush was used with each disk and therefore there could be some resultant variability in wear. The grooving seen is not uncommon with wear specimens. Once a groove begins to form it becomes accentuated as the test proceeds.

From the <u>in vitro</u> wear values seen in this study it appears that the polyether based materials, the Araldite mixture and 10 minute setting E-POX-E putty would be more suitable at resisting wear in the longer term. It would also indicate that TERM wears significantly less than Cavit.

# 6.6 Effect of Ledermix paste and Pulpdent paste on set materials

The only observable effects of either paste on the materials tested was that of surface staining by the Ledermix cement. For all materials other than Permadyne there was no significant effect on hardness when tested.

Due to the small sample size and the lack of a significant softening effect between the three test conditions for the other polyether based materials the results for Permadyne should be regarded with caution.

All materials were 24 hours old before testing commenced which does not occur

clinically where the materials would be in contact with the medicament pastes during the setting reaction. This may affect the materials physical and chemical properties unless separated by some other medium such as a cotton pellet.

The reason for testing for effects of medicament is that camphorated monochlorophenol (CMCP) an intracanal dressing often used in the USA has been shown to have a softening effect on IRM (Olmstead et al., 1977). In this study Ledermix paste and pulpdent paste did not appear to soften any of the test materials.

#### 6.7 General discussion

All the polyether based and epoxy resin based materials provided satisfactory seals in all substrates. The only traditional temporary restorative material to achieve this was Cavit. The performance of Cavit in the dimensional stability and wear tests was far poorer than that of all the experimental materials indicating that these other materials may be preferable, if found suitable for clinical use.

TERM sealed satisfactorily in all substrates other than amalgam and performed significantly better than Cavit in the wear study. This would indicate that where amalgam does not form a part of the cavity margin TERM should be used in preference to Cavit. Permadyne and the Araldite aluminium oxide mixture showed significantly less wear than TERM again indicating that these materials would be preferable.

The polyether materials could also be used in preference to a cotton pellet in the base of the cavity to act as a second barrier to leakage. The presence of medicaments &in the canal should prevent then from penetrating into the canals and possibly obstructing them.

When using traditional temporary materials such as Cavit and TERM a more satisfactory long term seal may be attained by using a double seal where a material showing good sealability is placed below a harder wearing material occlusally.

The poor performance, in this study, of traditional materials must be considered in light of the wide clinical acceptance and use of these materials. The bactericidal nature of some of these materials may have some bearing although bacterial microleakage studies have shown that all the materials used commonly show leakage after 3 weeks (Lamers et al., 1980; Keller et al., 1981; Blaney et al., 1981). Certainly the longer a seal is required the higher will be the risk for failure due to contamination by microorganisms. It is for this reason that improvements to current materials and techniques should be sought.

The non traditional materials examined in this study performed well in the tests described, often showing a more desirable behaviour than the traditional materials tested. Although questions need to be answered about biocompatability and the ability to perform during clinical loading, these materials warrant further investigation.

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# SUMMARY

It was the aim of this study to assess the suitability of dental materials not customarily used as restorative materials, and some epoxy resin based industrial materials, as long term temporary endodontic coronal sealing agents, and to compare them with materials currently in use.

This was to indicate which of a series of materials might show promise for further investigation and modification, rather than to develop a new material.

Tests performed were those which were thought to have greatest relevance clinically and were carried out sequentially as a sieve with materials being discarded from further investigation if they failed to perform adequately.

The only materials shown to have setting times greater than that which would allow full setting prior to the patient leaving the operatory were Cavit and 2 hour setting E-POX-E putty.

IRM and Ketac-bond performed very poorly in microleakage studies on tooth and were discarded from further investigation.

TERM sealed well in all substrates other than amalgam where only 20% of specimens sealed adequately indicating that it should not be used if amalgam forms some

part of the access cavity margin.

All other materials tested (Cavit, Genesis, Ramitec, Impregum F, Permadyne, 10 minute E-POX-E putty, 2 hour E-POX-E putty and Araldite 1:2 with aluminium oxide powder) sealed adequately in all substrates.

Cavit and Genesis showed most of their linear expansion to take place in the first 24 hours. All other materials except 2 hour E-POX-E putty showed a significant increase in expansion to 14 days.

Permadyne showed the least wear but was not significantly less than the other polyether materials, 10 minute E-POX-E putty and Araldite. TERM showed significantly less wear than Cavit which showed the greatest wear of all materials tested.

Ledermix paste and Pulpdent paste had no effect on the hardness of set materials. Ledermix paste induced surface staining of all materials.

The polyether and epoxy resin based materials tested in this study have performed well in comparison to traditionally used materials and subject to further investigations, such as biocompatability and mechanical cycling studies, may be shown to be suitable as endodontic access cavity seals.

# **APPENDIX I**

#### **MANUFACTURERS INFORMATION**

10 Minute E-POX-E Ribbon Putty, Loctite corporation, Conn., USA.

Alpine tooth polishing brush, Amalgamated Dental, London, England.

Application-Syringe, ESPE Gmbh, West Germany.

Araldite 5 Minute epoxy resin adhesive, Ciba-Geigy Ltd. Basle, Switzerland.

Cavit, ESPE Gmbh, West Germany.

E-POX-E Ribbon Putty, Loctite corporation, Conn., USA.

EM cutting Disk, Von Moppes-IDP Ltd., UK.

Etalon vernier calliper, Roch, Switzerland.

Genesis, Caulk/Dentsply International Inc., Milford, Del., USA.

Heath Schlumberger Strip Chart Recorder System EU-205B, Heath Company, International Division, Michigan, USA.

Impregum F, ESPE Gmbh, West Germany.

IRM CAPS, (Intermediate Restorative Material), Caulk/Dentsply International Inc., Milford, Del., USA.

Ketac-Bond Caps, ESPE Gmbh, West Germany.

Ketac-Silver, ESPE Gmbh, West Germany.

Ledermix paste, Lederle Pharmaceuticals, Wolfratshausen, West Germany.

Permadyne, ESPE Gmbh, West Germany.

Permagum, ESPE Gmbh, West Germany.

Pulpdent paste, Caulk/Dentsply International Inc., Milford, Del., USA.

Ramitec, ESPE Gmbh, West Germany.

Screw micrometer eyepiece, Vickers Instruments, London, UK.

Shore A durometer, Swick, West Germany.

Slipicone silicone releasing agent, Dow Corning Australia, Pty., Ltd.

SZ Steriomicroscope, Olympus Optical Co., Ltd., Japan.

TERM, (Temporary Endodontic Restorative Material), Caulk/Dentsply International Inc., Milford, Del., USA.

Translux CL, Kulzer & Co., Gmbh, West Germany.

Ultramat high velocity amalgamator, Southern Dental Industries, Australia.

Vitrabond, Dental Products Division/3M, St Paul, USA.

Wild M 400 Photomakroskop, Wild Heerbrug Ltd., Switzerland.

Zircate Prophy Paste, Caulk/Dentsply International Inc., Milford, Del., USA.

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