



SOME ASPECTS  
OF  
POSTERIOR RESIN RESTORATIONS:  
AN  
IN VIVO  
AND  
IN VITRO STUDY.

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## APPENDIX A.

## ESSAY:

## ASPECTS OF POSTERIOR RESIN RESTORATIONS

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## ASPECTS OF POSTERIOR RESIN RESTORATIONS

### 1. INTRODUCTION

The last 35 or so years of dentistry have seen some rapid and dramatic changes in the restorative field. This has been particularly evident in the non-metallic restoratives; i.e. the resin-based restoratives.

Synthetic restorations of this kind started with the unfilled acrylic resins in the early 1950's. The original aim of these materials was to overcome the aesthetic and biological problems of the older silicate cement restorations. However, it soon became evident that the resins had a number of disadvantages of their own. The first resins, the unfilled acrylics, had poor colour stability, readily-stainable surfaces and margins, and major marginal percolation; the latter due to significant polymerisation contraction and thermal cycling. Added to this, the early resins were not bonded to enamel or dentine, and had low hardness and stiffness. The percolation rate due to the marginal gap led to a high incidence of recurrent caries. Unlike amalgam, where corrosion products might "block" the marginal gap, the resins had no corrosion products to do this. Thirty-six percent of unfilled acrylic fillings were replaced for reasons of aesthetics or recurrent caries within five years, Smales (1977).

### 2. DEVELOPMENT OF THE MODERN RESINS

#### 2.1 Early Development.

Research and clinical use of the acrylics continued through the 1950's and 60's. Inorganic fillers were added to improve mechanical properties including stiffness and to reduce polymerisation contraction. Other

changes occurred in the polymerisation initiators with the use of sulfinic acids and mercaptans to improve colour change.

In 1955 Buonocore developed an acid etch system in an attempt to improve the clinical performance of the acrylics. By creating a bond between the enamel and the resin he hoped to produce a new system for fissure sealing. The system did not attract great attention until the late 1960's when it was applied to the BIS-GMA resin systems.

In 1956-7 the work of R.L.Bowen of the U.S. National Bureau of Standards produced the 2,2-bis[4(2-hydroxy-3-methacryloxypropoxy)phenyl]propane or more easily the "BIS-GMA" resin complex. This system has formed the basis of most modern restorative resins. The BIS-GMA complex is a compromise between epoxy and methacrylate resins, with the resulting complex being viscous and involatile.

The resin research over this period has evolved around the basic aim to produce the "perfect resin restorative" both for anterior and posterior use. The research areas centered on the composition of the resin itself, the coupling of the resin matrix to the filler content, the nature and quantity of the filler fraction and the different needs of composite resins for different applications. The resin restorative or "composite resin" as it is more commonly called was defined by Bowen in 1972 as:

"A combination of two chemically different materials with a distinct interface separating the components and having properties which could not be achieved by any of the components acting alone."

The basic ingredients in composite restorative materials include:

- monomers,
- polymerisation stabilisers,
- colour stabilisers,
- polymerisation initiators,
- polymerisation accelerators,
- inorganic reinforcing fillers and
- coupling agents.

## 2.2 The Traditional Resins

Following the transition period of silicates/unfilled/filled acrylics the evolution of resin entered what has been called the "Traditional Period". This era was the beginning of the widespread use of the BISGMA and later the Urethane dimethacrylate composite resins. Originally these systems did not have an enamel bonding system. However, they were initially hailed as a major improvement over their acrylic cousins. In the area of physical properties alone the traditional composites are far superior to both the silicates and the unfilled resins. However, widespread clinical acceptance soon gave way to wariness as a number of limitations became evident. The resins demonstrated poor aesthetics (only one shade was available), had poor marginal adaptation, left a dull, rough, plaque-retentive surface when polished, did not adhere to tooth structure, were occasionally lacking in form stability and lost anatomic form. In addition, there were other problems with marginal staining, porosity, lack of antibacterial action, no fluoride release, and finishing difficulties. The size of the filler particles was a major problem, the size ranging from about 8 to over 50 microns.

Thus these resins also became known as the "Macro-fills" due to the relatively large size of the filler particles.

The addition in 1973 of the acid etch technique, where a low viscosity unfilled resin was applied to the enamel after it was etched with ortho phosphoric acid to facilitate the adhesion of the composite resin to enamel, was perhaps one of the greatest single improvements in the use of restorative resins. However application of the resins remained very much as an anterior restorative. Their use in Class III,IV and V cavities was very common, although a number of people tried to use them in posterior teeth in Class I and II applications. The high wear rate due to loss of filler particles, as a result of resin matrix breakdown, did not lead to their widespread use as a posterior restorative. There was also a significant problem due to undetected recurrent caries associated with the lack of ability to maintain marginal seal and lack of radiopacity for easy detection. Inability to withstand occlusal loads is also a major problem with this group of resins.

### **2.3 The Microfill Resins.**

In an attempt to rectify some of the problems of the earlier resins changes to the resin matrix and more notably the filler content began to take place. To improve handling characteristics changes were made to the viscosity of the matrix by adding lower viscosity resins or diluents. The low viscosity monomers which were added included triethyleneglycol dimethacrylate (TEGDMA) and ethyleneglycol dimethacrylate (EGDMA). Other oligoethyleneglycol dimethacrylates, such as DEGDMA and Te-EGDMA are present in trace amounts. The addition of these diluents did improve the handling characteristics but at the expense of some of the physical

properties such as an increase in polymerisation shrinkage. The full details of these effects will be discussed later. The other major change to come was in the filler content, the beginning of the "Micro-fills".

The Micro-fills appeared in the late 1970's. This group were designed principally to overcome the aesthetic and wear problems of the Macro-fills without sacrificing any of their previous gains. The filler particle size was of the order of 0.04 microns. This enabled the resin to be finished to a very high lustre. These resin systems also showed a very high rate for colour stability, 98% acceptable by 1980, Vanherle et al. (1985). This was accompanied by a greater resistance to wear by abrasion. However, marginal adaptation did not show any great improvement, and in fact has remained a problem for this group of resins, and the mechanisms for this failure will be discussed later.

Micro-fills were used in both posterior and anterior applications, and while remaining suitable for most Class III and V applications they did not appear to be suitable for Class I and II applications. This was due to a lack of form stability and a wear resistance which was still inferior to that of amalgam. Other problems concerning radiolucency, marginal leakage, recurrent caries, interdental contact areas, porosities, cleft formation and incisal and facial chipping still remained. The main problems with their use in the posterior region are wear at occlusal contacts and bulk fracture.

#### **2.4 The Hybrid Resins.**

Following these developments came the so called Hybrid resins. These resins are a combination of the Macro-fills and the Micro-fills with most of the better characteristics of the modern versions of its predecessors and

with superior wear resistance. This development has led to an upsurge in the development of posterior resin restoratives and with it a general consensus concerning the basic criteria for the ideal posterior composite resin. These materials should possess the following general characteristics, Vanherle et al. (1985) and Jendresen (1985):

1. Biocompatibility
2. Wear resistance like amalgam
3. Ease of manipulation with simple conventional working techniques
4. Absolute form stability
5. Perfect and impermeable marginal adaptation
6. Radiopacity
7. Quick, exact and non-tooth-destructive finishing procedures
8. Good aesthetics

### 2.5 Curing System Developments

Further to these developments there is also the change in the way resin restoratives are now cured. Originally the materials were cured chemically where the system usually consisted of two parts, a resin base and a catalyst. Most systems use liquid/liquid for unfilled resins (usually the bonding resin) and paste/paste for the filled resin. This system had a number of disadvantages including limited working time and a higher rate of incorporated porosities. However, these systems do have an even cure. The newer resins have photo activated curing systems. The first of these systems used ultra-violet light and the more recent systems (1978 onwards) visible light from around the 470nm waveband, although they react to a significantly wider wave range. This system for curing the resins has a number of advantages including great control over working time. It has its

own set of drawbacks as well, most notably the depth of cure in the resin when it is activated by the light. These problems and others to do with the polymerisation process will be discussed in greater depth later.

### **3. DISCUSSION**

The following areas will be discussed in more detail and as they apply to posterior resin restorations.

#### **3.1 RESIN MATRIX**

#### **3.2 FILLER SYSTEMS**

#### **3.3 POLYMERISATION**

#### **3.4 COUPLING AGENTS**

#### **3.5 RADIOPAQUE SYSTEMS**

#### **3.6 PHYSICAL PROPERTIES**

#### **3.7 MECHANICAL PROPERTIES**

#### **3.8 CLINICAL TECHNIQUES**

#### **3.9 FAILURE MECHANISMS**

### **3.1 THE RESIN MATRIX**

#### **3.1.1 Function**

The resin matrix has three important functions. By definition the matrix provides the framework for the body of this type of restoration. It supports and holds the filler particles which are used to provide additional strengths to the restoration. Finally, in the unset phase it allows the restoration to be adapted to the irregularities of the cavity and occlusal form.

### 3.1.2 Monomer Combinations

As noted in the introduction, the basis for nearly all modern resin restoratives is the BISGMA complex, a hybrid resin formed from epoxy and methacrylate groups. The creation of this resin achieved two important theoretical goals. It had a low polymerization shrinkage and a high molecular weight for strength and set in a reasonable time in the mouth. Other resins with the desired properties were known to exist but they had extremely long setting times. However, this new resin was highly viscous, the higher the viscosity the lower the polymerisation shrinkage, Cowperthwaite et al., (1981). Clinically this highly- viscous resin is hard to handle particularly in those materials which also have a high filler content, such as the posterior composites. To counteract this, manufacturers have added various low viscosity monomers, such as TEGDMA and EGDMA, have used the diurethane dimethacrylate UEDMA as the only monomer or have used various combinations of all three. The quantity and type of the various monomers determines the quantities of unreacted methacrylate groups following polymerisation, termed the "degree of conversion" , Ruyter and Sjovik, (1981). The results give a varied mixture of physical properties and there is usually a loss of some of the desirable physical strengths of the material for a gain in ease of handling and placement.

The overuse of small diluents, principally the dimethacrylate monomers, caused a number of problems through excessive polymerisation shrinkage. The stress and strains accompanying the hardening reaction in some cases caused defective enamel margins despite acid-etch enamel bonding, Jorgensen and Shimokobe, (1975).

## 3.2 FILLER SYSTEMS

The incorporation of fillers became necessary in order to make the physical properties of the resin restoratives resemble those of tooth structure as much as possible. To do this a high proportion of hard strong particles is required. The other important properties which needed to be looked at in filler selection were particle size and shape, radiopacity, thermal expansion and a good refractive index match with the resin matrix.

### 3.2.1 Early Fillers

The first filler systems were introduced as early as 1951. The first fillers in the composite resins were glass fibres, soda-lime glass beads, synthetic calcium phosphate, fused silica and various glass formulations. Other reinforcing filler materials that have persisted include certain lithium-aluminosilicate crystalline compositions, crystalline quartz, pyrogenic silica, barium aluminoborate silica glasses and various others. In some materials the filler has been of one type only and in others it has been a mixture. The type and quantity of the filler has an effect on the overall physical properties of the restoration. One of the often used fillers has been quartz although glasses are more widely used. It is chemically inert and has the right index of refraction. The disadvantages of using quartz are that it has a high coefficient of thermal expansion and that it is harder than most other fillers. The high coefficient of thermal expansion, (higher than that of the tooth crown) increases the risk of marginal leakage and the hardness increases the difficulty in getting a flat, smooth surface with most finishing procedures.

### 3.2.2 Current Filler Developments

As has been described earlier the evolution of the filler component went through three basic periods; the Traditional Composites, the Microfilled Composites and the Hybrid Composites. The hybrid composites are thought to offer the most promise as amalgam substitutes although the microfills also have some interesting characteristics which are useful for the posterior application of resin fillings. The hybrid composites have developed along three separate lines.

The first, the true hybrid composite, consists of an organic matrix + traditional macrofillers + microfillers. The second are the hybrid composites with microfiller based complexes: organic matrix + traditional macrofillers + microfillers + splintered prepolymerised particles. The third group are also hybrid composites with microfiller based complexes: organic matrix + traditional macrofillers + microfillers + agglomerated microfiller complexes. An example of the first group is P-30, of the second group is Finesse and of the third group, Visiofil/Visio-Dispers. Clinical trials in anterior applications have shown that the second and third groups do not demonstrate superior qualities when compared to their microfilled equivalents.

The filler system needs to be able to help provide the ideal characteristics to posterior resins. By having a range of particle sizes the resin can maintain good finishing characteristics, similar to the microfills, and maintain the best physical properties possible. For example the material, P-30, made by the 3M corporation has a graded particle range from over 50 microns (1%), to under 1 micron (22%). This enables the material to have an average particle spacing of 0.11-0.14 microns, compared to a macrofill

such as Concise, which has an average particle spacing of 0.39 microns. Such a situation means that the effect of polymerisation shrinkage in the resin matrix is drastically reduced and the physical strengths of the filler particles are more effective.

These materials tend to have greater compressive strength, modulus of elasticity, tensile strength and mineral content. In addition they have a lower water sorption, and coefficient of thermal expansion. The wear rate is also significantly less due to the incorporation of the smaller particles between the macro particles, having the effect of reinforcing the resin matrix.

### **3.2.3 Current Fillers - Microfills.**

Some manufacturers have chosen to use a microfilled resin as a posterior restorative. Like the hybrids there are a number of ways these materials can be formulated in order to achieve the desired properties.

#### **3.2.3.1 Homogeneous Microfills**

The first group, the homogeneous microfills have a silane coated particle size of 0.04 microns which gives the filler content a huge surface area and thereby limits the filler loading. It is not possible to achieve an inorganic filler content of 75-80% as with the hybrid and traditional composites, Lutz et al. (1983), in fact the loading is closer to 50%. This makes the limiting factor the resin matrix itself and this phase undergoes plastic deformation and fissure formation, so these materials are not ideal for posterior use.

### 3.2.3.2 Inhomogeneous Prepolymerised Microfills

The second group, the inhomogeneous microfilled composites with splintered prepolymerised particles generally show a wear resistance superior to that of the traditional composites of the 1970's when used in stress-bearing posterior restorations. The breakdown usually occurs in the occlusal contact area and has been attributed to the effects of strong attrition forces and chemical disintegration. The problem appears to be that the bond between the matrix phase and the prepolymerised particles is not ideal because the heat curing of the prepolymerised part leaves few reactive double bonds which results in a low but sufficient rate of copolymerisation with the organic matrix, Lutz et al., (1983).

### 3.2.3.3 Spherical Prepolymerised Microfills

The third group are the microfilled composites with spherical prepolymerised particles. In this group the spherical prepolymerised particles have a surface which is permeable to the monomers of the organic phase. After polymerisation of the composite the spheres are completely incorporated into the matrix by polymer chains. It is believed that this group show some promise for both anterior and posterior applications.

### 3.2.3.4 Inhomogeneous Agglomerated Microfills

The final group are the inhomogeneous microfills with agglomerated microfiller complexes. An in-vivo study of these materials in MOD Class II restorations has shown them to be subject to hydrolysis. The agglomerated complexes were dissolved preferentially to leave large voids although the

surface wear resistance was close to the best microfills and hybrids, Lutz et al., (1983).

#### 3.2.4 Radiopaque Fillers

More recently a number of radiopaque formulations have been introduced. An example of this is one of the barium glass formulations which has the following basic composition:

SiO<sub>2</sub>, 66%

BaO, 17%

B<sub>2</sub>O<sub>3</sub>, 11%

Al<sub>2</sub>O<sub>3</sub>, 6 mole %, Bowen et al., (1972).

Such a formula has the following advantages:

- relatively low coefficient of thermal expansion,
- no monovalent elements and
- the proper refractive index.

However one its main faults is that it has an alkaline reaction in water due to soluble barium compounds at the surfaces of the particles. This affects the interaction with the silane coupling agents and as a result the filler particles may be poorly held which will lead to a loss of anatomic form particularly in areas of occlusal stress. Therein lies a major difficulty for the posterior composite restorative; on the one hand it needs to be radiopaque, particularly in the Class II situation and on the other, the very process of making it radiopaque may leave it with a serious deficiency to be able to withstand occlusal stresses and maintain anatomic form. Bowen et al., (1976b) have shown that it is possible to produce a radiopaque glass

filler which has been acid-etched to improve the binding between the filler particle and the resin matrix.

In the mid 1970's strontium was introduced to replace barium as the radiopacifier. This material has a much lower toxicity than barium and in the oxide form accounts for around 30% of the glass filler formulation. It has been noted that patients are often given a Barium meal without toxic effect.

### **3.3 POLYMERISATION**

#### **3.3.1 Introduction to Polymerisation**

The process of polymerisation is an attempt to cause various individual components to chemically bond together to form a single integrated body. In the case of restorative resins the reaction of constituent monomers to form a polymer provides a three dimensional organic matrix which supports the remaining components of the composite resin.

The degree of the polymerisation process and the degree of conversion within the resin matrix has a marked effect on the ability of that system to produce a viable long term restoration, for example, the quantity of remaining double bonds alters the tensile strength, Asmussen, (1982). The degree to which the resin polymerizes is dependent on several factors. These include; viscosity of the initial monomer systems, the rate of change to the viscosity that occurs during the setting reaction, (particularly in the initial stages), the presence of and the degree to which inhibitors affect the reaction, the system of activation for the reaction, and the amount of dilutant.

### 3.3.2 Initiation and Acceleration

Polymerisation, or curing, needs an initiator and an activator for the reaction to take place. These elements form free radicals which in turn cause the monomer units to bond together. Even the rate at which these radicals are formed will affect the degree of polymerisation. G. M. Brauer (1981), has listed a set of guidelines for the ideal initiator-accelerator system. These are as follows:

1) to generate sufficient concentration of free radicals with a minimum of radical wastage,

2) to produce enough free radicals to obtain an adequate working time for the monomer-polymer mix and a required curing time for the specific application is obtained, (not a great problem with VLC systems ),

3) to produce a concentration of free radicals to yield a polymer of a molecular weight distribution which will result in optimum physical properties of the dental restorative,

4) to form no undesirable by-products such as aesthetically unpleasant or colour unstable materials,

5) to be tasteless, colourless, non-toxic, non-irritating and completely biocompatible,

6) to use constituents that are storage stable for extended periods under the environmental conditions that might be encountered in transit, in storage, in dental supply houses or in the dental office, especially in tropical climates,

7) to incorporate ingredients that are readily synthesized at a reasonable cost,

8) to employ constituents that blend with, dissolve in, or are fully compatible with all components of powders, liquids, or pastes of resins or composites.

### 3.3.3 Curing systems.

There are two methods used in clinical dentistry to cure the resins. The original system, which is still used in a wide variety of applications, is chemical activation. More recently, photo activation, by either ultra-violet light or visible light has been employed and is now the most widely used system for the dental restoratives.

#### 3.3.3.1 Chemical Curing

Chemical curing uses an initiator, benzoyl peroxide, which is activated by N,N-bis(2-hydroxyethyl)-p-toluidine to produce the free radicals. These radicals then cause the monomers to bond, forming chains and randomly linked branches, which produce the three-dimensional polymer. The extent to which the reaction then proceeds depends on the availability of free radicals and free monomer to add to the developing polymer, the ability of the components to diffuse through the liquid/gel matrix, the presence of inhibitors, the quantity and type of filler used and the molecular structure of the monomers used. (Presently a wide variety of dimethacrylate monomers are in use.)

The chemical cure of the resins tended to be relatively even throughout the body of the restoration and the polymerisation contraction caused the

restoration to shrink inwards, producing stresses within and along the margins of the restoration, Hansen,(1982). This led to the term "contraction gap" which is the microscopic gap at the margin that causes microleakage between the restoration and the remaining tooth structure. The open surface of the restorative tended to shrink inwards and this helped reduce the shrinkage from the margins, particularly when an acid-etch bond technique was used. Where adequate bond strength is achieved, fracture of the adjacent enamel can take place due to the tensile stresses produced by this polymerisation contraction.

#### 3.3.3.2 Photocuring

Photocuring was introduced in the mid 1970's. The first systems used ultra-violet light ( "UVLC" ) as the activator to produce free radicals. These systems used long-wave ultra-violet radiation of about 365 nanometers (nm). In the late 1970's the visible light systems ( "VLC" ) began to appear. As previously mentioned, visible light around the 470 nm wavelength was employed. Resin systems which make use of photocuring can provided optimum working time and maximum opportunity for good wetting of the resin to the tooth surface, Phillips, (1981).

The UVLC systems use a photoinitiator such as benzoin methyl ether, which undergoes photofragmentation to form free radicals which in turn initiate the polymerisation reaction, Craig, (1981). The VLC systems use diketones and a reducing agent to initiate polymerisation. The diketone is usually camphoroquinone and the reducing agent, N,N-dimethylaminoethyl methacrylate, Craig, (1981). It has been assumed that the light energy causes the diketone to react with the reducing agent to produce an excited

state complex (exiplex) which then breaks down to give the necessary excited free radicals.

When free radicals for any of the systems are produced, they are not immediately available to the monomer for polymer production. The presence of dissolved oxygen which reacts more readily with the radicals produces an inhibiting effect. The time this effect lasts is directly proportional to the amount of dissolved oxygen in the resin, usually about 60 ppm. This is also the reason why unpolymerised surface films exist on uncovered resin during and after polymerisation. The atmospheric oxygen acts as an inhibitor. The thickness of this surface layer is dependent on the rate at which free radical formation occurs, the faster the rate, the thinner the layer. Thus the system which has the highest rate of energy available for activation produces the thinnest film. The UVLC system produces energy at a higher rate than the VLC system and both produce energy for radicals at a higher rate than the chemical curing system. Prolonged exposure in the UVLC systems have been shown to be advantageous in hardening the restoration throughout, Salako (1979) but, this has remained controversial.

The UVLC systems have fallen into disfavour. The reasons appear to include occupational safety to both users and patients and the inability of ultra-violet light to penetrate enamel. Furthermore the UVLC systems have a relatively shallow depth of cure, Salako et al., (1979), by comparison with the VLC systems. The main reason for this appears to have been the ratio between filler particle size and wavelength, Salako et al., (1979). The particle size was about half the wavelength of the UV light, a ratio which causes maximum scatter leaving vastly reduced amounts of UV light to

transmit into the resin to produce the necessary energy for the conversion reaction. Mitchell et al. (1986) have produced results from a study between two resins where the only difference was that one was activated by U.V. light and the other, visible light. The results tended to show the visible light cured system to be more durable, with better anatomic form and marginal integrity.

This situation leaves a paradox. The UVLC systems, due to the energy availability per quantum produced a higher bond conversion rate than VLC systems (about 70% compared with 62% respectively) but, as a result of filler interaction, had a lower depth of cure. This simply means that with ultra-violet light surface layers cured well but, with an abrupt fall off at a shallower depth. Results have been reported for Class I & II UVLC restorations which have shown relatively good results for occlusal loading, Wilder et al., (1984). However, Mitchell et al., (1986) suggest that visible-light-activated composite resins will yield more durable posterior restorations than the ultra-violet-activated composite resins.

The VLC systems give better results for depth of cure but as previously mentioned, they have a lower conversion rate (62%). The conversion rate is higher in VLC systems than chemically-cured systems, Asmussen, (1982). The optimum conversion rate is dependant on the resin system and the type and condition of the light source used. Those lights emitting a component of ultra-violet light do however give better overall conversion. One of the major drawbacks of the VLC resin systems is that a number are sensitive to dental operating lights. Most modern operating lights have quartz halogen globes similar to the VLC curing lights. The material cured this way has a poor conversion rate and is not to be relied upon. Direct sunlight has

excellent curing properties requiring only 10-15 seconds, compared with an average 40 seconds per increment from a VLC light.

Polymerisation does not finish when the curing light is removed. Hansen, 1983, has reported that conversion continues for some time with an increase in surface hardness being the main indicator of this process. The greatest change occurs in the first few minutes with detectable changes still occurring at 60 minutes.

### 3.3.3.3 Special consideration: DEPTH of CURE.

A considerable amount of work and discussion exist around this topic. Following the clinical use and observed failures of some UVLC applications there have been many articles written concerning what constitutes a cure, what affects the cure, and what we consider to be the proper depth of cure. Finally there are the problems of relating the observed in vitro behaviour to the actualities of the clinical situation.

Numerous factors have been observed as having an effect on depth of cure. Considering both the in vivo and the in vitro situations, these include the following:

- mould material,
- mould size,
- exposure time,
- photo activating light source intensity,
- diameter of the light beam,
- age and condition of the light globe,
- condition and type of light transmitter between the source and the objective, and

inherent factors of the composite itself, principally the transmission coefficient and the composition.

The method for measurement of depth of cure has not been standardised, and to date several systems have been tried. These include scraping, dye adherence, deGee et al., (1984), optical distinction, hardness indentation profiles, and physical measurement by micrometer. There is also no consensus about absolute hardness value or the relative ratio of hardness between the top and bottom surfaces for a depth of cure criterion. Skeeters et al. (1983), defined depth of cure as the axial distance from the surface where the Knoop hardness dropped to less than 90% of the hardest value measured. The specimens for this definition were cylinders of composite material, 4 mm in diameter and 6.4 mm high. The specimens were produced in cylindrical split/brass moulds. During curing, the tip of the light source was placed in contact with a Mylar matrix strip that covered the base of the specimen cylinder.

There is little evidence to show that prolonged exposure to the curing light will eventually provide adequate depth of cure. The manufacturers suggest that if in doubt a longer exposure time should be used. Leung et al. (1982) did demonstrate that extremely long exposure times (300 seconds) will improve the polymerisation. However, they used top and bottom Barcol hardness measurements to gauge adequate depth of cure.

When considering the previously-mentioned factors some reasons for discrepancies can be seen. For example where the beam diameter is less than the internal mould diameter the depth of cure increases because the surrounding composite acts similarly to a translucent mould, Fan et al., (1984). Translucent moulds have often been cited as producing higher

depth of cure values when compared with opaque metal molds. However, because metal moulds behave in a similar fashion to tooth structure their use has been substantiated, de Lange et al., (1980), Watts et al., (1984) and Ruyter, (1985).

The transmission coefficient is the indicator of the ability for the curing light to pass through the composite. The composition of the material will affect this value. For example, the small filler particles of the microfills cause more light scattering, which decreases the amount of light transmitting through the material giving a shallower depth of cure and meaning that the microfills generally have a lower transmission coefficient.

With regard to variations in curing lights, investigators generally agree that this is an area where considerable variations can occur. A number of articles, Blankenau et al., (1983), Watts (1984), Friedman et al., (1984), Fan et al., (1985), Cook (1985), and Clinical Research Associates, (1986), have attempted to rank different light sources according to their ability to provide adequate depth of cure. The rankings between the reports often vary and several reasons for this have been given and they include:

- differences in measuring methods used and the instrumentation,
- variations in the relative performances of identical units, (i.e. units of the same brand ),
- variation in globe output,
- method and condition of the light transmission medium to the composite as previously mentioned,
- diameter of the emitting surface of the light source, and
- distance of the light source to the composite.

Globe output can vary with the age, subtle variations in the manufacture, or line voltage variations to the globe at the time of measurement.

The output of the lights in the 400-500 nm range also varies and it is this property that has more bearing on the effectiveness of the unit rather than brightness as 470 nm is the optimum wavelength for free radical formation. Currently not enough is known concerning the quantum efficiencies of the various wavelengths for curing. The observation is that VLC units with higher quantities of ultra-violet light as well as high output in the 470 nm region are more efficient for curing. The actual light intensity recorded from a given unit is therefore not always an accurate indicator of its efficiency. Manufacturers have been increasing the radiant emittance of the lights in order to overcome this problem, Cook, (1985).

Several studies have shown specific problems associated with depth of cure. In 1984 deGee, ten Harkel-Hagenaar, and Davidson concluded from their dye staining of unreacted groups in the resin phase, that microfill resins have a more limited depth of cure than conventional resins, that the boundary between separately light activated layers in built up composites occasionally resulted in defective curing, and that in deeper areas of some light-activated microfills insufficient coupling between the prepolymerised filler particles and the matrix can be demonstrated.

The 1980 work of de Lange, Bausch and Davidson demonstrated the presence of three zones in photo-initiated composites. The first, zone A, showed physical properties comparable to those of the chemically-cured composites. The second, zone B, demonstrated an increased softness

compared to zone A, and the third, Zone C showed no signs of cure at all. These results were based on hardness values derived from Knoop hardness indentations. The tests also confirmed the work of Reinhardt and Vahl (1977) which showed that only unfilled resins had a time-dependent curing pattern. Other points that arose from their work are that it is possible that a second exposure after finishing the restoration has only a slight effect on the total penetration depth, and that during finishing of a fresh photocured restoration, the best material (surface layer) as regards bond conversion rates may be removed.

A number of other reports have demonstrated that factors such as shade variation will have an effect on the depth of cure. The darker shades of several resin systems have been shown to require at least 40 seconds in order to achieve adequate hardness and presumably depth. Also darker shades show a definite loss in desired physical properties between 1 and 2 mm and little or no cure beyond 3 mm depth. This has led to recommendations of incremental build-up for deep and extensive restorations. However a more recent study suggests that the relationship between shade and depth of cure may not be all that straight forward and that the depth of cure may be more dependent on other factors such as the translucency of the components, e.g. opaque shades, Swartz et al., (1983) and Ferracane et al., (1986). Furthermore, the discrepancies between different lights tends to be more pronounced when their ability to cure darker shades is compared, Friedman et al., (1984).

The position of the restoration within the tooth and accessibility for the curing light also have a marked effect on the depth of cure. This is particularly important in Class II cavities with regard to the interproximal

region. It has been shown that increased area of light transmission and lights of greater output can increase the hardness values for the resins, particularly the posterior composites, Watts et al., (1984). These researchers also demonstrated that the curing profiles of a given material were different when activated by different light units.

Finally, it has been shown that over-exposure of the material to some of the modern dental operating lights adversely affects the final properties of the restoration by premature curing, particularly during the manipulative time of placement. The complete extent and exact nature of this detrimental effect is not known.

#### 3.3.4 Effects of Viscosity

The viscosity of the constituents also has a major influence on the polymerisation and resultant conversion rate in composite resin systems. The high molecular weight monomer BIS-GMA ( molecular weight 512 ) is also the most highly viscous component of the resin matrix. This gives it the lowest polymer shrinkage of all the most commonly used monomer and oligomer systems (6.4%). The average percentage of reacted double bonds is only 56% which when compared with methyl methacrylate (98.6%), is relatively low. TEGDMA on the other hand has a polymer shrinkage of 12.5% but a higher percentage of reacted double bonds (68.2%) (MW 286 ). For most of the commonly used polymers the polymerisation shrinkage is inversely proportional to the molecular weight of the monomer. An exception to this is neopentyl glycol dimethacrylate, NPGDMA, (MW 240), which has a highly branched nature, and produces a relatively-low crosslink rate relative to TEGDMA, (15.6% vs 38.5% respectively ), but has a similar shrinkage rate to TEGDMA (12.2% vs 12.5% ). This means that the

shrinkage in the highly crosslinked network is dependent on both the molecular weight of the monomer and the efficiency of the utilisation of the double bonds in a crosslinking mechanism. Therefore, by using a diluent monomer such as TEGDMA to improve the viscosity of the resin system an increase in polymerisation shrinkage can be expected, as well as an increase in crosslinking and reacted double bonds. An improvement in the resulting physical properties occurs at the cost of increased shrinkage in the resin matrix. This can then be offset to a degree by the nature and amount of the inorganic filler content of the composite resin. Ferracane et al. (1982) suggest that the improved crosslinking provided by the use of diluents is reflected in an improved tensile strength.

The other area where viscosity has been of concern is the depth of penetration of restorative resin into acid-etched enamel. Asmussen, (1977) concluded that the viscosity as such was not a limiting factor for the penetration of resin monomers into the etched pores of enamel and that the rate of dissolution of air into the monomer may determine the depth, thus affecting the ultimate tag length.

### **3.4 COUPLING AGENTS**

#### **3.4.1 Coupling Requirements**

Researchers have long realised that optimal performance of the composite resin systems is dependent upon a good bond between the organic resin matrix and the inorganic filler content. In order for a composite restorative to have substantially improved mechanical properties, transfer of stress under loading from the high strength, dispersed reinforcing

filler to the more ductile polymer matrix must occur, and this requires such a bond.

There have been two approaches to this problem. The first was to rely on a mechanical interlocking between the matrix and the filler, and the second was to produce a chemical bond between the two.

#### 3.4.2 Mechanical Interlocking

Mechanical interlocking relied on irregular shaped particles and was therefore not very effective when spherical particles were employed. A porous irregular structure could be produced by either sintering fibres or particles together, or by etching away a continuous phase of glass. These composites tend to have a lower modulus of elasticity when compared to chemically-bonded systems. Unbonded filler fractions also have a marked reduction in physical properties when very high filler loadings are used.

#### 3.4.3 The "Silanes"

The chemically bonded systems first appeared in the early 1960's. Today the bonding agents belong to a group commonly called the "Silanes", which is an abbreviation of "organofunctional silane coupling agents". The most often used agent is  $\delta$ -methacryoxypropyltrimethoxy silane, A-174.

The silane coupling agents exist in water solution in hydrolysed form as a triol with the fourth silicon bond linked to a reactive organic structure. These silane triols adsorb on the filler surface in monomeric or oligomeric form. Subsequent drying completes the condensation process linking the coupling agent molecules to each other and to the filler surface by siloxane bonds. These bonds involve hydrolysis of the methoxy groups with either

bound surface water on the reinforcing filler or silanol or aluminol groups of the filler. The unsaturated carbon double bonds are then available for polymerisation with the matrix during the curing of the composite, Soderholm, (1985).

A number of researchers have commented that the silane does not form a uniform layer but is found as granules or micelles which are joined by thin continuous films or channels of low molecular weight material. The silane films appear to be thicker than monolayers and the granules appear to contain high density polymer. The lower density films and channels appear to offer an easy path for the entry of water which causes hydrolysis of bonds holding the various components together. This along with other factors which affect the quality of the silane coverage, have a major influence on the lasting strength of the restoration. These other factors include the surface geometry of the filler, its composition, and the presence of foreign materials. Reinforcing fillers that produce an alkaline environment in water can degrade the attachment of the coupling agent, Craig (1981) and Soderholm (1985 p.153). Surface treatment of these fillers to deplete the surface of alkaline ions may improve their performance. Such fillers often contain barium or strontium salts which are known to produce alkalinity, but have been required to increase radiopacity, a major clinical requirement for posterior composite restoratives. Furthermore the optimum number of chemical bonds required at the interface for longevity of the restoration in the oral environment is not yet known.

The silane content of the resin system is only very small, as one gram of these substances can cover about 314 square metres of reinforcing filler surface, Craig (1981). The effectiveness of the silanisation of the filler

particle surface has been under considerable investigation. Improvements have been reported where addition of n-propylamine ( a promoter ) enhances the silanisation of A-174 to silica surfaces in normal aliphatic hydrocarbon solvents. It has also been reported that where the solvent is cyclohexane a more water resistant silica-silane bond results. The result is an improvement in the diametrial tensile strength of the final composite resin.

The extent of the chemical bonding of the silane to the filler depends on the silanisation system used. In the absence of the promoter, the silanisation reaction is said to be very inefficient, particularly where aromatic solvents are used. The quality of the silane treatment greatly affects the durability of the composite particularly when modified glasses are used for the filler, Erickson et al. (1986 ).

#### 3.4.4 Copolymeric Bonding

Where prepolymerised particles have been added to increase the volume fraction of the filler there is a further chemical bonding system between the filler and the matrix. This is the co-polymeric or homopolymeric bond between the organic matrix and the partially organic filler. Such a bond relies on the availability of unreacted double bonds in the prepolymerised particles and the ability of the monomer to diffuse into those prepolymerised particles.

### 3.5 RADIOPAQUE SYSTEMS

#### 3.5.1 Radiographic Requirements

One of the most important clinical characteristics of the posterior composite restoratives is their ability to project a radiopaque image. There are a number of reasons for this. They include the ability to detect recurrent caries, check for overhangs and restoration contour, detect voids, and to be able to see overall outline clearly distinct from the normal tooth structure and surrounding tissues.

Radiopacity has been achieved by the incorporation of elements with relatively high atomic weights into the glass fillers. A large number of the heavier elements are not suitable because of the colour they impart to the glass. Lead is not suitable because it causes discolouration through the formation of sulphides.

#### 3.5.2 Barium Glass

A further problem with the incorporation of heavy elements is the possibility of an adverse change in the refractive index of the filler particle. Barium is available in commercial glasses but with a higher refractive index than is desirable. Work done in 1972 by Bowen and Cleek produced an experimental glass filler with 17% barium oxide as one of its constituents. This glass became the forerunner to a new series of radiopaque fillers. The main problem with these materials is their inherent alkalinity which leads to leaching of the barium constituents and hydrolysis of the polymer. An effective silane coating of these particles which sealed them from water would prevent this. The soluble barium compounds are also known to be toxic, although it is questionable whether

enough would escape from the composite and migrate to the pulp to cause irritation or would diffuse into systemic areas to cause harm.

### 3.5.3 Strontium Glass

The toxicity question has led to further research with modified oxides such as strontium oxide which have lower toxicity. The manufacture of such glasses does have some problems where the components can separate at elevated temperatures. However it is possible to use this element for its radiopacity and because the refractive index ( 1.55 ) is sufficiently close to BISGMA. Strontium behaves in a similar way to calcium both chemically and biologically, which accounts for its reduced toxicity. Calcium oxide has also been used and although it forms good clear glass it does not have sufficient radiopacity to be clinically ideal for posterior composites.

### 3.5.4 Effects on Glass Phase Morphology

One of the other major limiting factors in the use of heavy elements in the formation of these radiopaque glasses is their ability to develop suitable interconnecting phase morphology with the range of phase dimensions present in the glass formulation. Unsuitable connections render the glass susceptible to acid and other chemical attack and various components are then leached out and the composite can then break down, Bowen and Reed, (1976a and 1976b). The first indications of this are discolouration by staining of the leached particles held on the surface of the restoration.

### 3.6 PHYSICAL PROPERTIES

The typical physical properties of composite resins as listed by Craig in 1981 are as follows:

3.6.1 Polymerisation contraction,

3.6.2 Porosity,

3.6.3 Thermal coefficient of expansion,

3.6.4 Thermal conductivity,

3.6.5 Water sorption,

3.6.6 Water solubility,

3.6.7 Diffusion coefficient on sorption,

3.6.8 Contact angle.

#### 3.6.1 Polymerisation contraction

As previously discussed, during curing the resin matrix undergoes contraction and the amount and type of monomers and oligomers have a direct bearing on the magnitude of the contraction. The filler content has little effect on this contraction unless it is greater than 50% by volume, and clinically this shrinkage produces the so called "contraction gap". With most modern composites this contraction can be expected to be around 2-3%, by volume. However, with very low viscosity monomers such as methyl methacrylate it can be as high as 5%. The lowest value currently claimed is 0.6% by volume for the quartz filled posterior resin, "Visio-Molar", manufactured by the Espe Corporation.

#### 3.6.2 Porosity

There have been a number of reports concerning porosity, Fischel et al., (1982), Reinhardt et al., (1982), and Jorgensen et al., (1983). Those

concerning the chemically cured composites generally reported a porosity value of 1-2%, and that the porosity was greater when the materials were placed with an instrument than when injected, and that it could be markedly reduced by the application of pressure for a short time to the mixed composite, the extent of this reduction being a function of the viscosity of the mix.

The recent change to light-cured resins has altered the expected porosity a great deal. These materials are vacuum packed and can be delivered to the cavity without producing porosity, particularly when they come in the syringable form. The spatulation of the chemically-cured systems incorporated a number of porosities and this has now been eliminated. However, operator-induced porosity is possible through the mishandling of the VLC resins with hand instruments for placement where voids are created or bodies of air entrapped.

The use of VLC materials has led to only two essential variables for porosity formation. They are handling technique and the viscosity of the resin. As previously mentioned direct injection of the material into the cavity produces the least porosity, but it is still not porosity free. Only when the inside of the syringe was wetted with a drop of monomer before loading the composite did a porosity free delivery take place, Jorgensen et al., (1983). This may not be necessary for compules prepacked under vacuum. However it must be realised that manipulation of the syringed material by a hand instrument may still cause porosity formation as a result of entrapment of induced air bubbles.

It has been reported Medlock et al., (1983) that syringe placement significantly reduces the pore area of composite resins compared to hand

placement and that it can give total elimination of voids larger than 150 microns.

The placement of composite material without bonding resin has been studied by a number of researchers, Jorgensen, (1975), Asmussen, (1977) and Hansen, (1984). The main aim of most of these studies was to see and assess the ability of the filler resin to produce tags of material which could penetrate the etched enamel and effect a satisfactory bond, and in fact a number agree that this is quite possible. However such an approach appears to be prone to causing porosity, and the use of a bonding monomer reduces markedly any porosities that may occur in the region of the restoration margin.

Larger porosities in the region of cavity irregularities were more prone to occur when a bonding resin was not used, Hansen (1984). Also, the diameter of the opening of the manufacturer's syringe had some bearing on porosity formation, as it was seen in work by Hansen (1984), that the larger the diameter the lower the rate of porosity. Furthermore, his work demonstrated that it was probable that polymerisation of the bonding resin prior to placement of the filled restorative would adversely affect the ability of the bonding resin to help reduce porosity.

### 3.6.3 Thermal Coefficient of Expansion.

The coefficient for normal tooth structure is from 10 to 15 X 10<sup>(-6)</sup> per degree C. Macrofilled composites have a value of about 26 to 40 X 10<sup>(-6)</sup> / °C and the microfills have values from 46 to 70 X 10<sup>(-6)</sup> / °C . The difference between the two types of resin is related to the larger amount of organic phase in the microfills. However, the more difference between the resin

systems and the tooth structure means that thermal cycling will produce a range of varying expansions between the two, and marginal leakage will result. Resin systems with larger volumes of filler (>50%) have a reduced thermal coefficient of expansion.

Due to the differences in expansion and contraction between the enamel and the restorative material there is additional stress placed on the acid-etched bond. As microfilled resins have a thermal coefficient of expansion which is two to three times greater than the conventional composites, the cyclic effects of constantly changing temperatures can lead to earlier bond failure and material fatigue, Craig (1985 p.230). Tensile stresses produced during thermal expansion could facilitate crazing and crack growth within the matrix of composite resins. This effect is more pronounced in resins with higher filler fractions and is thought to offer a reason for good clinical wear resistance seen in microfilled composites, Soderholm, (1984).

#### **3.6.4 Thermal Conductivity**

The composite materials compare very favourably with both dentine and enamel in this area. This applies to most of the composite restoratives currently in use. However there are differences between the various composites. Microfills have a lower conductivity because the colloidal silica filler has very low thermal conduction. Composites which use quartz have values almost three times higher than the microfills due to the high conductivity of that material.

#### **3.6.5 Water Sorption**

The problem of water sorption in composites is a constant area of concern as any water which may enter the material can hydrolyse bonds to cause

breakdown of the resin matrix. The more highly filled and greater the viscosity of monomer the longer it takes and the more difficult it is for water to enter the resin system. Also the greater the degree of crosslinking the lower the uptake of water. The microfill resins have water sorption values which are between three and six times greater than the new posterior restoratives, Craig (1985).

The other major clinical result of water sorption is expansion of the composite over a period of time until equilibrium is reached. This reduces the effects of polymerisation shrinkage due to a degree of compensation in the opposite direction. Equilibrium has been shown to occur after varying periods greater than 14 days, Craig (1985).

#### **3.6.6 Water Solubility**

Composite resins have shown slight solubility, in the order of 0.05 mg per square centimeter. The effects of solubility will be discussed in "failure mechanisms". The majority of detectable solubility in the early stages after polymerisation, is the loss of residual monomer remaining after the reaction. Following this, leaching of inorganic ions can be detected, where such ions are the result of interfacial bonding breakdown, or first, from the cut filler in the polished surface.

#### **3.6.7 Diffusion Coefficient on Sorption**

The diffusion coefficient for composites in water is about  $1.1$  to  $3.1 \times 10^{-9}$  cm<sup>2</sup> per second compared to a figure of about  $16 \times 10^{-9}$  for poly(methylmethacrylate).

### 3.6.8 Contact Angle

The contact angle gives an indication of the wettability of the composite and is of significant importance as it influences the marginal leakage of the restoration. The advancing contact angle of water on composites is about  $65^{\circ}$  and so the composite is classed as a hydrophilic solid. With this contact angle and a contact angle for tooth structure of about  $55^{\circ}$ , water or saliva will spontaneously penetrate any crevice, (e.g. contraction gap) between the restoration and the tooth. The hydrophilic surface is also more likely to absorb any precursors to plaque or stains.

Researchers have shown that the driving force for capillary penetration along a contraction gap is a negative change in the free energy at the solid/ liquid interface. If the free energy change is positive, pressure is required to force the liquid into the capillary space. The theory is to produce composites which exhibit a contact angle greater than 90 degrees, i.e. hydrophobic composites, to reduce or prevent water penetration. Experimental composites that demonstrate hydrophobic properties have been produced, for example, "Adaptic II".

## 3.7 MECHANICAL PROPERTIES

### 3.7.1 Types of Properties Considered

The development of the composite resin has to achieve, as one of its aims, mechanical properties which will allow it to behave in the same manner as the tooth structure it is replacing. A typical listing for mechanical properties appears below:

Compressive strength (MPa)

Compressive modulus (GPa)

Diametral tensile strength (MPa)

0.1% Yield strength (MPa)

Transverse strength (MPa)

Shear strength (MPa)

Elastic modulus (GPa)

Poisson's ratio

Modulus of resilience

Fracture toughness

Hardness

Indentation depth

Recovery from indentation

Wear

As there are such a large number of listed properties some of which are not very meaningful to clinical dentistry, brief comments will be made for some, while others will be discussed in greater detail.

### 3.7.2 Compressive Strength

The compressive strength of the posterior composites tends to be higher, (241-400 Mpa), than the conventional composites, (186-297 Mpa ), which in turn have higher strength than the microfills, (172-228 Mpa ). The new quartz filled resins have an ultimate compressive strength approaching that of the high copper amalgams, (Dispersalloy, 387 Mpa), Craig, (1985).

### 3.7.3 Tensile Strength; Diametral

The posterior and conventional resins exhibit similar values for this property, (34-76 Mpa ), which are significantly greater than for the microfills, (28-34 Mpa), Craig, (1985).

#### 3.7.4. Shear Strength

This property exhibits similar behaviour to tensile strength, the conventional and posterior composites being better than the microfills. In all cases both tensile strength and shear strength are much less than compressive strength. However, although this implies that composites are not brittle materials, i.e. that the relatively soft resin controls the ultimate compressive strength, brittle rather than ductile failure has been shown to occur, Fan et al., (1979).

#### 3.7.5 Modulus of Elasticity

This is a very important clinical indicator as it is believed to give an indication of deformation under masticatory forces. The modulus is dependent on the composite being a bonded system, (i.e. matrix bonded to filler ). The use of different size distributions of filler particles in the posterior composites has allowed a filler content in the order of 80% by weight which appears to be the threshold for great increases in the elastic modulus.

A high elastic modulus clinically means lower deformation in the restoration and surrounding tooth which leads to less microleakage and reduced risk of cusp fracture.

#### 3.7.6 Modulus of Resilience

The modulus of resilience is a measure of the energy absorbed in the elastic range of the materials. Microfills, which react in a similar way to unfilled acrylics under elastic deformation, exhibit similar values to the more conventional composites, but the elastic modulus is quite different. For this

to be the case it means that these materials allow more strain to occur before permanent deformation occurs. This is why the microfilled resins are not as brittle as their more highly filled counterparts.

### 3.7.7 Fracture Toughness

The fracture toughness of posterior composites has been shown to be higher than that of usual occlusal loadings as catastrophic fracture rarely occurs in posterior composites, Draughn, (1985). Loss of filling substance is more likely to occur gradually over a longer period of time.

The work of Lloyd and Iannetta (1982) has demonstrated that the addition of filler to polymer to produce a dental composite improves the stress intensification factor which they also demonstrated to be a good indicator of fracture toughness. They were also able to show that submicron filled composite was not as tough as composite with larger particle sizes, such as the small particle size hybrids (1-5 micron range ). The small particle hybrid composites with very heavy filler contents, as used in posterior resin restorative materials, have an inherent advantage over other composites for fracture toughness, Lloyd and Mitchell, (1984).

Another factor demonstrated as having a major influence on the toughness is the presence of water in the composite. The absorbed water has a plasticising effect which is sufficient to effectively aid molecular movement in the matrix which dissipates energy. This effect decreases as the water sorption value of the composite decreases, Lloyd, (1982).

Temperature has been demonstrated to have an effect on fracture toughness but it is not significant over the temperature range anticipated to exist over any length of time in vivo.

Lloyd, (1983), concluded that posterior composites are brittle materials and that fracture toughness was the best measure to determine resistance to fracture. The values of fracture toughness for posterior composites were similar to other composites with similar filler loadings and the materials were comparable to the high-copper amalgams, but not as tough as the low-copper amalgams. The final conclusion that deserves mention was that the fracture path was in almost all cases through the matrix.

### 3.7.8 Wear

The problem of wear in composite resins has occupied the minds of innumerable researchers. It is a major concern for the success of posterior composites. Many attempts have been made to produce an in vitro system to measure wear of candidate posterior composites, which will give an accurate indication of how they perform clinically. However, it has been realised by a number of researchers that a proper and accurate wear test may take as long as a proper clinical trial to carry out, and that to accelerate such tests may reduce the relevance of the test, McKinney, (1985). The evidence to date suggests that the wear itself is a product of abrasion, fatigue, adhesion loss at the resin filler interface, and chemical disintegration, and that during wear there is a buildup of subsurface damage.

#### Wear mechanisms:

##### 1. Occlusal contact areas

Fatigue, plus adhesion loss, plus abrasion

Chemical disintegration

##### 2. Contact-free occlusal areas

Abrasion

### Chemical disintegration

### 3. Interproximal contact areas

#### Abrasion

#### Chemical disintegration

Factors which influence the wear of the posterior composite are; the nature and composition of the resin matrix, the filler loading and type, the effectiveness of the filler-matrix coupling, and the ability of the composite to resist the environmental factors in the mouth. The oral factors include the relationship of the restoration to the occlusion, the nature of the diet including abrasiveness of food particles and solubility parameters of ingested organic liquids, and other changes such as thermo-cycling and pH shifts.

In 1978, Draughn and Harrison pointed out that wear or more particularly abrasion rates are dependent on the size, hardness, and volume fraction of particles in the composite, and that the most abrasion-resistant composites available at the time, contained a high volume fraction of large, hard particles.

#### 3.7.9 Hardness

The hardness of light-cured composite resins appears to change significantly after irradiation. Leung et al. (1983), reported significant increases after both 10 and 20 minutes, with smaller increases observable after 7 days. The magnitude of increase was related to the initial degree of polymerisation. However, polymerisation was judged by top and bottom Barcol hardness readings.

In 1985, Kanca reported that although it may seem desirable to use hardness values to judge posterior composites, it may not be the only aspect to be considered when making such comparisons, because of the different filler particle composition and loading. Kanca further suggested that the uniformity of cure would be a better avenue for comparison.

### 3.8 CLINICAL TECHNIQUES

The dental profession has for many years used silver amalgam as an effective and tolerant restorative material for posterior restorations. This material has demonstrated servicability despite clinical abuses during mixing and placement. It has been a relatively simple and very versatile material to place in a number of clinical applications. Certainly, no one can doubt that the optimum performance of amalgam is achieved in conservative cavities where placement has occurred by the use of the best clinical techniques, but even so amalgams do stand up reasonably well to contamination and other indiscretions which may occur during its insertion.

The posterior composites have demonstrated a number of problems when used in Class I and II cavity preparations and these problems will be described during the different stages of the clinical technique. The primary indication for the use of composites is in premolars and molars where appearance is a major concern. The other problems with amalgam fillings include corrosion at the margins, causing ditching, on the surface resulting in tarnishing, staining of the surrounding tooth and fracture. Secondary issues concerning the possible toxicity of mercury from amalgams have caused emotive reactions for their use. However, this is still an area of major controversy and as yet the posterior composites are not

able to perform as well as amalgam for the larger Class II restorative situations. Where the occlusal stresses are judged to be large, then these materials are not indicated.

The clinical techniques for these materials will be discussed under the following headings:

3.8.1 Tooth Isolation

3.8.2 Cavity preparation

3.8.3 Pulp protection

3.8.4 Matrix bands

3.8.5 Adhesion to the remaining tooth

3.8.6 Placement of the restorative

3.8.7 Finishing

### 3.8.1 Tooth Isolation

Following anaesthesia and prophylaxis of the tooth to be restored, the area is now isolated. This is preferably done with the application of rubber dam. Any moisture contamination during the procedure will only reduce the optimum properties of the finished restoration. Therefore, the best seal possible with the dam is required, including inversion of the dam at the cervical margins around the teeth protruding through the dam in the operative area.

The inversion helps in two areas. First, it helps retract the gingival papilla interproximally and second, it keeps the crevicular fluid seepage under control.

The next step is to pre wedge the tooth if proximal surfaces are involved. This allows slow separation of the teeth to facilitate the proper formation of

contact points interproximally. Wedging can also reduce seepage by blanching the papilla and reducing the blood flow, and displaces the rubber dam while protecting it during cavity preparation.

The most vulnerable aspect of the posterior composite restoration is microleakage at the interproximal gingival margin. Good moisture control and access to this area during the operation are therefore a prime concern and hence the pointed reference to isolation and separation.

### 3.8.2 Cavity Preparation

The aims of cavity preparation are to remove the carious lesion and to restore the tooth as near as possible to the original condition. Posterior composites offer an advantage in this area over the amalgams because they can be bonded directly to tooth structure. This allows for more conservative cavity preparations and the possibilities of actually reinforcing the damaged tooth structure during restoration, by the binding effect produced by the bond.

The preparation should at all times be as conservative as possible. Any remaining anatomical ridges should not be removed, sharp cavity outlines and sharp line-angles are to be avoided. Occlusal contacts are not to coincide with cavo-surface margins. The interproximal margins need only very minor clearance for finishing and the cavity walls in this area should be parallel to the enamel rods. The nature of the cavosurface margin does remain an area of controversy. A number of clinicians believe that there should be a 45 degree bevel of 0.5 to 1.0 mm length to facilitate the best adhesion for the optimum marginal seal. Support for this comes from the many articles which demonstrate that adhesion is stronger to transversly

cut enamel rods than to parallel enamel rods. Beveling has also been shown by Oilo and Jorgensen (1977), to reduce the number of observed enamel fractures at the cavity margins. An increase in the bevel size further reduces the incidence of enamel fracture.

However, the opponents of this theory have some equally valid points. They argue that during the course of cavity preparation a significant amount of the enamel is already cut obliquely and that further beveling is unnecessary, Boyde, (1985). In addition, it has been demonstrated that these margins may be prone to fracture and leave a ledge-type defect which, amongst other things, produces a plaque trap. The final argument against the bevelled margin is that it is difficult to finish as the margin often becomes obscure. This is particularly evident when there is little or no difference between the shade of the tooth and the restoration, and can lead to excessive removal of sound enamel.

Simonsen, (1985), suggests that the use of a bevel on the occlusal surface may not be indicated as this increases the surface of the composite resin exposed to wear and that a cavity preparation made parallel to the long axis of the tooth will provide a bevel. Simonsen does feel that bevels in the interproximal area are required.

Wilson et al. (1986), concluded that in their ongoing study there was no significant difference in the performance of butt-joint and bevel-edged margins.

Where an existing amalgam is being replaced the cavity is generally larger than necessary for conservative posterior composite restorations, and so all reasonable care is required to minimise any further tooth destruction. The

only preparation that need apply, save that to remove recurrent caries, is to ensure that margins are accessible and out of direct occlusal contact if possible.

### 3.8.3 Pulp Protection

The protection of pulp vitality is of paramount importance and it is particularly vulnerable during acid etch procedures. Traditional concepts require a calcium hydroxide lining of the exposed dentine and any direct pulp exposures. More recent concepts suggest Ledermix cement for direct pulp exposures, and glass ionomer cements as linings and bases. Results from such regimes are certainly more than favourable.

A further consideration in pulp protection, is possible toxicity due to the diffusion of remaining ingredients from the cured composite restoration. It is quite probable that the remaining uncured resin components could have an effect on the underlying tissue for a very long time, Spanberg et al., (1973). While only limited research has been done in this area it has been shown in histological studies that inflammation of the pulpal tissues occurs in response to the presence of composite restoratives. It has also been concluded that inadvertant etching of the dentinal tubules, removing any plug and opening them will promote this reaction. The ability of the bonding agents and/ or lining materials to prevent this has not been completely investigated, although a new technique for screening dental restorative materials for chemical toxicity to the pulp has been described, Hume, (1985).

### 3.8.4 Matrix Bands

The use of a matrix in the Class II situation is mandatory. The purpose is the same as for an amalgam, however, the application and type of matrix is much more critical. Due to the predominant use of VLC systems for posterior composite resins and the need to get as much light as possible to the resin to effect the cure, clear plastic matrices are advocated by the resin manufacturers.

Metal matrices have been used in this application for a number of years. The advantages of these systems are that they are generally thinner than their plastic counterparts, can provide better contours, and are easier to negotiate through the contact area. A more reliable gingival margin adaptation can also be created as burnishing produces a permanent change in the contour of the band, whereas the plastic matrices show a tendency for memory and spring back to their original position. This applies to all areas where contours are to be guided by the shape of the band. The big disadvantage with the metal matrix is that it is not possible to shine the curing light through it.

The plastic matrices as mentioned above have some problems. The difficulties encountered with contour are important as this means that some areas need to be contoured mechanically after removal of the matrix. This presents a new area of skill required by the dentist as it is easy to damage adjacent sound enamel while trying to produce an adequate contour. The time consumed in performing this operation is far greater than simply carving back an amalgam with a hand instrument. The incidence of excess material and flashing is very high and this adds to the finishing problems.

The major advantage of the plastic materials is that the VLC materials can be cured from the interproximal direction. This is very important as the curing shrinkage of the material is in the direction of the light source, and to minimise the possibility of shrinkage straining the bond at the gingival margin, the material is in effect cured as well as possible towards that margin. Special light-transmitting wedges have also been developed to assist in this gingival region. The design of these wedges allows the light to transmit through the length of the wedge and be redirected at right angles to the wedge towards the gingival margin by an internal reflector. The ultimate effectiveness of this arrangement is yet to be seen due to the infancy of this recent innovation.

#### 3.8.5 Adhesion to The Remaining Tooth

The adhesion of posterior composites to remaining tooth structure has been a matter of major concern. The prevention of microleakage by good adhesion is tremendously important for the prevention of recurrent caries which has been a major downfall of composites in posterior applications, Horn, (1981). The bonding systems available include the "Dentino-Enamel" type bonding agents such as "Scotch Bond" and "Bondlite" and the use of glass ionomer cements and filling materials which are acid etched and have the restorative composite bonded to them, McLean, (1985). The use of ferric oxalate has also been reported to improve bonding to dentine, Dumsha, (1984).

The technique currently employed during this study involves the use of glass ionomer as both a base and a dentine bonding agent. Such a system has a number of advantages. The glass ionomer is bland to the pulp unless the cavity is exceptionally deep. The material is also anti-cariogenic due to

the slow release of fluoride, and after the removal of the smear layer by a 10 second application of citric or polyacrylic acid it binds to the dentine with adhesion at least as good as the dentine-enamel bonding agents. This gives the clinician an ideal base to support the posterior composite. It also provides gingival marginal seal when the glass ionomer is used as the restorative material for the first millimeter or so in the gingival portion of the proximal box.

Following the application of the glass ionomer lining/base the tooth is left so that the material can mature before standard acid etching, McLean, (1985), and Mount, (1986). This is a disadvantage as filling materials used as a base such as Ketac-fil take 15 minutes to attain a maturity which allows high enough bond strength to the composite, Mount, (1986). These materials also are at a disadvantage as they are not radiopaque. The material Ketac-bond, may be an easier material to use as it attains sufficient maturity in about 5 minutes, and it is radiopaque. While these materials are setting they are sealed with the composite bonding agent as any drying or moisture contamination will severely affect their physical properties. After the maturation time the bonding agent is removed and the acid etch is applied. The etch is usually carried in a gel medium to allow accurate placement with minimum attack on structures not directly involved.

Standard etching principles apply, using ortho-phosphoric acid around 37-40% concentration for about 60 seconds. The etchant is then removed by the application of a water/air spray for about 30 seconds and the tooth is then thoroughly dried. The etch is only placed on the required enamel surfaces and the glass-ionomer base. Accurate application can be

facilitated by the use of a standard tuberculin syringe and fine needle to inject the etchant only onto those areas required. Erickson and Glasspoole, (1986) and Andreaus and Heymann, (1986), report no significant difference between the use of gel or liquid etchant on the final bond strength, provided the times for etching, washing and drying remain the same.

The enamel-dentine bonding agent is then applied, taking care not to disturb the fine crystals exposed to provide the mechanical interlock. Excess material is carefully removed, usually by air blowing. The bond can then be cured, but if left it provides an excellent lubricant for the placement of the posterior composite material.

The nature of successful dentine bonding has been a cause for a lot of research with attention being given to a number of different types. The two main systems available at present bond either to the inorganic phase of the dentine, e.g. Scotchbond and Bondlite, or to the organic phase (collagen) in the case of Gluma. Both Gluma and Scotchbond have been shown to reduce the contraction gap between restorations and the tooth, but the degree of success varies widely between researchers, Finger (1986). Retief et al., (1986), noted that, in their study and those of others, the magnitude and range of bond strengths obtained in vitro suggest that the clinical application of the bonding agents may give unpredictable results. Bonding at the cervical margin has been shown to be relatively unsuccessful, with Torstenson and Brannstrom (1986) stating that the use of pretreatment and dentine bonding agents did not reduce the gap size markedly. Asmussen et al., (1985), reports that bond strengths of 5 MPa produce marginal gaps of the order of 10 microns and that a bond strength of about 20 MPa is required to produce gap free fillings in cavities

involving dentine. Davidson et al., (1984), pointed out that the competition between the development of the composite-dentine bond and the contraction stresses of polymerisation will determine the success of that bond.

Laser technology is currently being investigated as a pretreatment to both dentine and enamel prior to the application of the bonding resin. The effect on dentine appears to be superficial, creating melted caps of dentin on top of only partly melted material. The polymer bond to "lased" dentin was found to be very strong. The effect on enamel appears to be greater, leaving a surface roughness which is sufficient for polymer bonding and which is also more resistant to acid dissolution, Nelson et al.,(1986), and Jongebloed et al., (1986).

### 3.8.6 Placement of The Restorative

The final placement of the actual filling material has several parameters which, based on the previous discussion, must be observed. The considerations to be observed are; the shade of the material, the proximity of the restoration to a gingival margin, the position of the pulp and the depth of the cavity, the position of the cavity within the tooth, the direction from which the curing light is applied, and the way in which the increments of resin are to be built up. Two other considerations exist, the choice of instrument to manipulate the resin and the type of delivery system to be used to get the resin to the cavity.

As has already been indicated, where the resin will be in close proximity to the gingival margin, it may be desirable to use glass-ionomer filling materials in the base of the proximal box because of their adhesion,

compressive strength, and anti-cariogenic abilities, rather than to rely on the dentino-enamel bonding agents. The use of glass-ionomer cement as a base also protects the pulp from the possible toxic effects of uncured resin which may exist from incomplete curing if the factors alluded to in the discussion on depth of cure are not adhered to.

Darker shades and deep cavities necessitate the use of small increments to build up the body of the restoration. The further the increments are from the exit window of the light source the greater the effect of the inverse square law as regards the light intensity. Increments should be placed obliquely in the proximal box and the curing light positioned so that on activation the curing shrinkage is towards the gingival and axial walls of the cavity. Only when the occlusal increment is placed should the curing light be applied from this direction. This regime for curing materials placed in Class II cavities should produce the least stress on the gingival margin, Vanherle, (1985), which is universally recognised as the most susceptible area for these types of restoration to develop failures. Bond strength between layers decreases with the age of the initial layer and follows the setting curves of the composites, Boyer et al., (1984). Eliades and Caputo (1986), have studied the bond strength between cured and uncured layers and report an increase in bond strength if the oxygen inhibited layer of the first increment is removed with acetone prior to placement and curing of a second increment. This is in conflict with most previous thought, where the oxygen inhibited layer was believed to act like a bonding resin for the next increment. The use of acetone is of limited clinical value.

Reduction of porosity formation can be achieved by using a material which is syringable directly into the cavity, Jorgensen et al., (1983), Millstein et al., (1984), and Hansen (1984). This method of delivery is also much less time consuming. Final manipulation to adapt the material to cavity walls and line angles should be done with a rounded plugger or ball burnisher which will comfortably fit into the cavity. Sharp instruments create bubbles and voids in the restoration. Lubrication of the instrument with a fine smear of bonding agent assists the placement and reduces further porosity formation, Hansen, (1984). Over-use of bonding resin for lubrication will lead to a marked reduction in the physical properties of the restoration because it disturbs the high filler : resin ratio of these particular posterior resin materials.

The techniques for placement of the resin restoration are still subject to disagreement between operators. The work of Finnegan and Wilson (1986), suggests only the use of hand instruments as a means of creating the least porosity. Placement techniques would appear to be dependant on operator variability and the handling characteristics of the particular restorative material as wide variations have been reported, and the trend is for manufacturers to produce syringable materials.

### **3.8.7 Finishing**

Considerable discussion has also been directed at this area, as reported by Lambrechts et al., (1982). The aim of finishing is to produce appropriate contour and to redevelop a surface texture that will reflect light in a similar manner to that of tooth enamel. The final result should also be resistant to surface staining and abrasion. The main methods include the use of stones, tungsten-carbide finishing burs, aluminium oxide-coated

flexible discs, fine and superfine diamonds, fine polishing pastes and rubber-mounted abrasives. The final finishing of composite resins involves gross finishing and contouring, fine finishing, and polishing. The time for final finishing needs to be considered, as it has been reported by various authors, Hansen, (1983), that the resins continue to polymerize after the curing light has been removed. Hansen also reported the importance of avoiding early contact with water where a possible detrimental effect may result.

In 1983, Lutz et. al. demonstrated that fine and superfine diamonds (40 and 15 microns respectively) produced a better finish on the four types of resin they used than did green stones, white stones, or tungsten-carbide burs. They also pointed out that the flexible aluminium oxide discs produced an excellent finish but were only useful on accessible convex surfaces. However, these researchers and others were quick to point out that as usual the best finish is obtained where the resin is polymerised against a matrix. But, this surface is usually rich in resin matrix and could present problems in an occlusal contact area. Where a matrix is not used, the outer layer polymerisation is inhibited, resulting in a chalky, soft surface. Therefore the removal of this layer to expose potentially harder material is advised by Lutz.

The 1984 report of de Wet and Hardwick points out that the rubber mounted polishing instruments can cause smearing of the surface, and could leave debris attached to, or incorporated in the surface. It was also pointed out by Pearson and Messing, (1979), that both rubber mounted finishers and discs produce considerable amounts of heat when being used and, with the relatively low melting temperature of the resin, smearing can occur. This

smearing covers the remaining exposed filler particles, which are left after the abrasive material chips away the surface. Where white stones and tungsten carbide burs are used the exposed filler particles are left protruding.

The work of Lambrechts and Vanherle (1982), has added further light to the effects of polishing. They point out that the smear layer exists in almost all composites they tested (14 altogether), and that it was caused by friction producing enough heat to melt and deform plastically, the matrix. The thickness of this layer can be as great as 100 nm. Another observation was that the finish of the hybrid materials was hampered by the unequal physical properties of the matrix and filler phases. Furthermore, it was observed that the grinding or polishing agent had to be harder than the hardest phase in the composite, and that these very hard and strong particles should not shatter but stick very well to the polishers. Loose abrasive grains tend to roll over the composite surface instead of grinding it, causing the organic matrix to be worn away and roughness to appear. The loose particles also fill up any air bubbles and pores and any subsequent polishing removes these particles causing further scratching. Their final conclusions were that it was better to finish for a longer period with a fine grained disc than to go faster with a coarse disc which loses bigger grains and that all loose particles must be removed from the surface immediately and continuously because it is the bigger particles of former polishings that drift over the surface and prevent complete polishing by the ultra-fine grains. The micro filled resins polish better than the conventional resins, Kanter et al., (1983).

More recent work by Smith et al., (1986) and Quiroz et al., (1986), demonstrated rubber-mounted abrasive points in the form of Shofu Quasite Midi-points and Vivadent polishing points to be the most effective in producing a smooth finish in posterior composite restorations. Considerable difference between the different brands of finishing systems were also detected. One brand of finishing diamonds was significantly better than the other tested. The same result occurred between the two disc systems tested, and the two rubber point systems.

Savoca and Felkner (1980), demonstrated that smoothness of composite resin after finishing is not a function of the time at which it is finished.

### 3.9 FAILURE MECHANISMS

All restorative materials used in dentistry have had some form of failure and posterior composites are no exception. Based on the previous discussion it can be seen that failure occurs in a number of aspects related to the nature, handling and clinical requirements of the posterior composite restorative systems. The areas attributing to these failures will be discussed under the following headings:

3.9.1 Wear

3.9.2 Microleakage

3.9.3 Water Sorption

3.9.4 Polymerisation defects

3.9.5 Physical defects

3.9.6 Clinical difficulties and miss-handling

### 3.9.1 Wear

Most writers in recent times agree that posterior composite restorations do not show the same ability to withstand wear as amalgams, Phillips et al., (1973) Leinfelder et al., (1975), Kusy, (1977), and Leinfelder, (1981), although the more recent hybrid resins do show an improvement over the traditional composites which were first used for this purpose, Pallav et al., (1986). The wear of posterior composites involves the breakdown of the resin matrix and its bond to the filler particles, and the eventual loss of those particles. This can occur from direct mechanical stress, or by chemical breakdown of the resin matrix, Leinfelder, (1981). Water plays an important role in wear, where stress corrosion and increased hydroxy ion concentration at the filler interface cause the loss of filler, which explains in part why composite materials do not withstand wear and tend to change colour with time, Soderholm, (1983).

Factors involved include the abrasiveness and chemical nature of the diet (e.g. frequent contact with alcohol increases the rate of breakdown of the matrix), the presence of occlusal interferences and bruxism, the method for finishing the surface, and the type of restorative material used with particular reference to the filler content and the matrix formulation.

Until recently, surface wear was measured using the United States Public Health Service system which tended to suggest that the wear rate appeared to accelerate between the first and second year where occlusal stress was involved, Leinfelder, (1985). However more recent work has shown that two thirds or more wear occurred during the first six months and that the relatively low rate initially observed in the USPHS system was due to the inability of the operators to detect the cavo-surface margin until it was

exposed by as much as 100-150 micrometers, Leinfelder, (1985). Most researchers now agree with the fact that in vitro wear tests do not necessarily concur with in vivo tests, and can not be used to give an indication of long-term in vivo performance, Powers et al., (1983). Goldberg et al., (1984), demonstrated the inability of the USPHS system to establish statistically significant differences between composite formulations at three-, nine-, eighteen-, and twenty-four month recalls. The technique of ranking models identified a statistical difference between the formulations at two years and the categorizing procedure, Goldberg et al., (1984), identified significant differences at nine, eighteen, and twenty-four months, Goldberg et al., (1984). Yost et al., (1986) pointed out that where sufficient serial specimens are available differences between materials can be seen, and wear rates estimated.

The work of Jorgensen, (1980) has demonstrated some interesting aspects concerning the relationship of the resin formulation and wear. First, he points out that the abrasion of the matrix in a macro-filled composite will not occur when the distance between the particles is of the order of 0.1 microns or less, and he feels that although this phenomenon is not completely understood, it may be related to the corpuscular or cellular nature of human food. This work also pointed out that as wear did occur with these resins, the increased friction produced by added surface-roughness led to a greater degree of surface abrasion and an increased wear rate. Porosity of the resin also enhanced the wear rate, Jorgensen, (1980).

When the surface of a composite restoration is periodically observed by scanning electron microscope the filler particles can be seen to become

more and more exposed from the resin and eventually they are lost, leaving microscopic holes and exposing the matrix to direct abrasion. The work of Abell et al., (1983), estimated the rate of recession of the matrix to be from 0.08 to 0.16 microns per day in a quartz-filled composite in a class I cavity.

Toothbrushing has also been implicated in composite wear and the study of this factor has demonstrated some important information concerning the effects of abrasion on the surface of various types of composite, Aker, (1982). The hybrid materials generally were seen under SEM analysis to have surfaces similar to the traditional composites after abrasion, although some were smoother than others. The surface characteristics were more attributable to the larger filler particles. The microfilled resins tended to retain a smooth surface. Both the hybrids and the microfills tended to be more abraded than the macrofills, the difference being up to six times, Aker, (1982).

Chemical exposure to 1.23% APF gel increases the risk of wear. The gel has been shown to produce surface roughness and weight loss, Kula et al., (1983). The filler particles are suggested to be the most likely sites for degradation but unpolymerised materials are also known to leach out. Phosphoric acid and hydrofluoric acid are constituents of the gel, Kula et al., (1983). The effects differ according to the filler type, with the strontium glass-filled resins being most affected, followed by the quartz-filled resins, and least affected are the silica filled resins, Kula et al., (1983). Variations in the size, type, and percentage of filler particles can alter the severity of these effects.

In 1982 McKinney and Wu reported that chemical softening by ethanol was far more effective than water in producing increased wear in dental

composite. They also observed light-cured materials to be initially more resistant than chemically-cured materials, and wear increased rapidly with decreasing surface hardness. Wear rates for strontium-glass-filled composites pre-conditioned in pure water were noticed to be enhanced considerably when compared to quartz-filled and micro-filled composites. In this situation the degradation was attributed to stress corrosion of the glass filler, McKinney and Wu, (1985).

The work of Lutz et al., (1984), demonstrated some important features concerning wear. They were able to show that vertical loss of substance in occlusal contact areas was more than double that in contact-free areas, and that the larger the restoration the greater the wear. This suggests that the resin restoration benefits from the protection offered by the surrounding enamel and therefore the cavities should be kept as conservative as possible. Curing mechanism was shown to influence wear also. Heat-cured resins had the least wear, followed by light-cured, and the greatest wear was associated with chemical-cured resins.

There have been various reports that the microfilled posterior resins have shown very little wear. The work of Asmussen and Jorgensen (1982) has given a possible explanation, where microfills at relatively-low stress have demonstrated higher fatigue strength. Clinically this would be possible where the restoration was of a conservative nature with protection from the occlusion provided by surrounding enamel. However, microfills have also been shown to be the most stable material in a wet environment, with respect to crack formation. These composites leach less silicon than composites containing strontium and/or barium glasses, Soderholm et al., (1984).

Materials with urethane-dimethacrylate/dibutyl-phthalate resin systems showed inferior in vivo wear resistance in occlusal contact areas. This is thought initially to be a result of the measurable plastic deformations associated with these materials. Fractures from these deformations initiate the breakdown of the restoration. The inclusion of prepolymerised particles can help to provide an initial wear resistance.

The hybrid composites with a reduced average particle size (compared to the traditional composites), and sophisticated size distribution, more dense packing of their macrofillers, and direct admixture of microfillers to the organic matrix, are believed to have more wear resistance than the traditional composites, Lambrechts et al., (1985). Their surface morphology is also superior to the traditional composites, meaning that their abrasion resistance is better.

Lambrechts et al., (1985) point out that although there are five types of wear (abrasive, corrosive, fatigue, adhesive, and attrition), probably the most aggressive is attrition. Attrition is seen as the ultimate wear consideration as it takes into account the effect of fatigue wear in centric stops in posterior teeth. The long-term effects of thermo-mechanical fatigue and cyclic-stress have yet to be fully understood, as these effects only show any destruction much later, despite any early favourable wear results.

Despite the wear problems in the posterior region, the composites have generally shown good marginal adaptation.

### 3.9.2 Microleakage

The effects of microleakage have been of concern to the dentist for a long time. When composite resins cure they contract, and if there is insufficient strength in the bond between the resin and the tooth, the adhesion of the resin to the tooth fails and a gap forms between the restorative material and the cavity wall. The effect is to allow in bacteria and plaque. This results in secondary caries, pulpal sensitivity, and sometimes pulp infection and necrosis. Hygroscopic expansion of the resin in the first two weeks is often not enough to close this contraction gap.

The main area where microleakage is applicable in posterior resin restorations is the interproximal gingival margin, Kidd, (1985), where there is little or no enamel to provide a sufficiently sound seal. Normally, the acid-etch enamel margins provide considerable resistance to microleakage, Luescher et al., (1977) and Hembree (1980). Unless particular steps are taken to prevent leakage at the gingival margin the restoration will fail, as irrespective of the material used contraction gaps develop at the cervical margin. This gap varies in width from 7.1 to 21.9 microns and the use of dentine adhesive or recommended pretreatment procedures for bonding does not markedly reduce the gap size, Torstenson, (1986).

Sealing of the gingival margin can be achieved by the use of a post-operative sealing technique as described by Torstenson et al., (1985), where low viscosity resin is drawn into the gap by capillary force and allowed to set. The gingival margin can also be sealed by the use of dentino-enamel bonding agents, although their effectiveness is questionable if the curing shrinkage of the filled resin is allowed to cause a high tensile force on the

bonding agent. The use of a glass-ionomer resin as a base extended to the gingival margin can also provide sufficient strength for a seal.

Significant reduction of microleakage at the enamel margins can be achieved with a bevelled cavosurface margin, Moore, (1986). At the gingival margin this reduction can be improved by the use of oblique incremental placement techniques and light-transmitting wedges, Krejci and Lutz (1986 ).

### 3.9.3 Water Sorption

Restorative resin systems have all been noted for their water sorption. This factor, which is variable according to the different resin formulations, often has deleterious effects on the long-term properties and performance of composites. The effects of water sorption are:

- Hygroscopic expansion,
- Plasticising of the matrix,
- Hydrolysis of the matrix bonds,
- Hydrolysis of the coupling agent-filler bond,
- Leaching of filler particles.

The effect of hygroscopic expansion has been considered as a positive aspect because it counteracts the setting contraction to some degree, ranging from 0.07% to 0.80%, Jensen et al., (1985). However, the other effects of water sorption are detrimental and far outweigh this positive aspect. Hygroscopic expansion has been implicated in the surface degradation of composites, where considerable damage leads to reduced hardness and enhanced wear. Furthermore, internal stresses are created between the filler particles and the resin matrix leading to debonding

between these two elements. Cross-linking between the resin components is also affected. The result is an overall plasticising effect of the system with a decreased ability to withstand occlusal loading, Ruyter, (1985).

In vitro testing of one posterior composite demonstrated that the creep deformation when the material was saturated with water was much larger than for its dry control, Ruyter, (1985). The microfilled composites had larger creep values than the hybrid composites and it was seen that the higher the filler content and lower the water uptake, the smaller the difference in creep between wet and dry specimens, Ruyter, (1985).

Although hydrolysis of the matrix bonds does occur the effects of the hydrolysis of the bonds associated with the filler coupling interface are the first to be seen. This is because this bond is readily attacked and results in crack propagation along this junction, Soderholm, (1985).

The filler particles have different reactivities to water, although the overall leaching of ions does not actually reflect this. Strontium and barium glasses have higher reactivities than silicon dioxide, but because the latter is present in much higher amounts there tends to be a similar amount lost. Matrix swelling stresses the Si-O-Si bonds of the amorphous fillers such as quartz, changing the bond angles and allowing the water molecules to attack the bond, causing the loss of silicone. This is known as stress corrosion.

#### 3.9.4 Polymerisation Defects

The majority of posterior restorative resins today are cured by visible light. Unlike the chemically-cured resins which cure uniformly throughout the bulk of the material, the VLC restoratives are activated only to a certain

depth. The efficiency of this activation results in a degree of conversion of carbon double bonds to form crosslinks between the monomers to achieve the branched polymer matrix. The degree of conversion is usually expressed as a percentage and for most commercial posterior resins varies between 60 and 70 %. Other factors such as the shade, reflectance and refractive index will also alter the degree and depth of cure. Anything which adds to the number of pendant residual carbon-carbon bonds can make the polymeric matrix more susceptible to oxidative degradative reactions, Antonucci et al., (1983). Poor conversion can also add to plasticising effects within the composite, Ruyter and Osaed, (1981).

The presence of unreacted monomer and oligomers can also have a plasticising effect on the polymer, which will alter the physical properties and the clinical performance of the restorative material. The depth of cure will also affect the water sorption and solubility of the composite, with increases in both these aspects as the depth of the resin increases; presumably due to the lower conversion rate and the resulting increase in free monomer and oligomer, Fan et al., (1986)

Defects allow greater water sorption, leading to increased swelling and gap formation. This in turn leads to more fluid flow and filler particle breakdown and ion leaching. Incomplete bonding to the filler particles causes a greater loss of these particles during function. Loss of quartz particles is particularly bad due to the secondary abrasive effect. The glass particles tend to be bonded more effectively and tend to break up rather than be dislodged as a whole, creating a softer abrasive medium.

The presence of molecular oxygen has a profound effect on polymerisation, and inhibits the reaction to the degree that any resin surface in direct

contact with oxygen will not set. This is a useful advantage when bonding new increments of resin to those already cured and in a fresh state. However, the presence of oxygen in porosities will inhibit the polymer reaction.

During the initial course of polymerisation the propagating free radicals, the unreacted dimethacrylate molecules, and the pendant methacrylate species are free to diffuse throughout the system and interact readily. As the number of crosslinkages increases the monomers form polymeric chains of increasing length with numerous branches. This causes the system to gel and the individual components are no longer able to freely diffuse and react, resulting in an inhibition for further saturation of carbon bonds. This effect is known as "steric hinderance". The quantities of unreacted methacrylate groups in composite resins are determined in part, by the type and quantity of the various monomers due to their different rates of diffusion.

#### **3.9.5 Physical Defects.**

The presence of physical defects in posterior composite resins is particularly important because they are used in load-bearing restorations. The highly-filled composites have been shown to exhibit more stable mechanical properties, Papadogianis et al., (1984). In general they act like viscoelastic solids. The ability to withstand creep and to recover is important where continual occlusal loading takes place. The creep properties of dental composites under conditions of optimal conversion are influenced by the content and type of filler as well as the final structure of the organic phase, Ruyter et al., (1982).

The conclusions of Papadogianis et al., (1985), give some insight to the areas where physical defects are important. From the results of the composites they tested, the following conclusions were drawn:

- 1) Posterior composites exhibit linear viscoelastic behaviour at low deformations,
- 2) Posterior composites have higher shear moduli and are less susceptible to creep than conventional and microfilled composites,
- 3) The moduli of the posterior composites were less temperature sensitive than the conventional and microfilled composites,
- 4) The maximum shear modulus occurred at 48 hours following preparation in two cases, and one week for the third. Subsequent softening was attributed to water sorption, and
- 5) residual strain, observed 50 hours after removal of the stress, was highest in specimens stressed within 24 hours of preparation. Very low residual strain was observed in specimens 48 hours to 8 weeks of age when the load was applied.

The physical defects which will affect these parameters are:

porosity,

microstructure, (filler type and interparticle spacing ),

water sorption and stress corrosion,

cracks, and

polymerisation shrinkage.

Ogden and McCabe, (1986), conclude that porosity and air inclusions lead to a significant reduction in fatigue strength, which is likely to cause decreased wear resistance in vivo.

The most wear resistant posterior composite resins appear to come from the hybrid group as a general rule. However, it has been demonstrated by Tani and Nambu, (1986), that marginal fracture resistance was higher in the microfills than the macrofills, with the concluding comment that the marginal fracture toughness is influenced by the filler particle size rather than the filler content.

Montes. et al., (1986), conclude that water sorption lowers the strength of the interfacial bonds thereby reducing the effect of the silane coupling agents, and lowering the failure stress and elastic modulus. This makes it easier for crack propagation. Wet materials were shown to have a greater extent of interfacial failure.

The presence of cracks in the composite can cause failure of the restoration because the cracks will propagate when the restoration is put under load. The cracks maintain a predominant path through the matrix, only occasionally passing through a filler particle, Lloyd, (1983).

The polymerisation contraction of the resin matrix creates a number of problems, including induced stresses and contraction gaps. The magnitude of the volumetric contraction is dependent on the shade of the composite resin, the type of filler, the filler loading and the exposure time. The value for volumetric contraction varies between 1.4 and 3% according to Walls et al., (1986). These figures represent a small sample but are generally indicative of the posterior composites, although figures of over 5% have

been reported, Vanherle et al., (1985). The stresses induced are considerable and it has been reported, Feilzer et al., (1986), that the cavity wall can be shattered by the rapidly developing contraction stresses and the tooth can develop flexure of the cusps, which can be painful. This means further consideration must be given to cavity design and materials handling.

### **3.9.6 Clinical Difficulties and Miss-Handling.**

The posterior resin restoratives have a number of aspects which add difficulty to their use in Class I and II restorations. These are:

- 3.9.6.1 Lack of packing feel,
- 3.9.6.2 Slumping,
- 3.9.6.3 Flashing across the cavo-surface margins,
- 3.9.6.4 Lack of shade contrast with tooth material, and
- 3.9.6.5 Post-operative sensitivity.

#### **3.9.6.1 Lack of packing feel.**

The lack of packing feel in posterior composite resins makes it difficult to adapt the restorative to the walls and angles of the prepared cavity. This is likely to result in voids and lack of marginal adaptation.

#### **3.9.6.2 Slumping**

Slumping is a major problem with resin materials used in Class II cavities. This inadequacy makes it difficult to produce correct anatomical form and interproximal contact. The material has no resistance to change in shape after it has been placed and before it has been cured, and so may be

deflected by "back pressure" of the matrix band or simply not hold shape against gravity.

#### **3.9.6.3 Flashing across the cavo-surface margins.**

The flashing across margins has special implications for the Class II cavity restoration, because it is difficult to remove in the interproximal area, particularly where conservative cavity preparation has left the margin in an inaccessible area. This is an indication for pre-operative separation. The clinical significance of a retained flash lies in whether the flash is bonded to the enamel or whether it is separated by a layer of pellicle. The later case would allow microleakage and probably recurrent caries.

#### **3.9.6.4 Lack of shade contrast with tooth material.**

Shade contrast with tooth structure is important for finishing the restoration margins. It is often a problem to delineate between enamel and the composite resin, and therefore difficult to determine when sufficient material has been removed when trimming before final polishing. The result can often be inadvertant removal of sound enamel. To overcome this problem some manufacturers have produced restorative resin materials which are non-matching in colour to the enamel. The G.C. company have produced a coloured varnish, (dark green), which is painted over the tooth before cavity preparation and acts as a cavity outline marker.

### 3.9.6.5 Post-operative sensitivity.

Post-operative sensitivity has a number of causes. These include;

Etching of the dentine,

Toxicity of the composite resin,

Polymerisation contraction and microleakage,

Polymerisation contraction and cusp flexure, and

Deflection of the restoration under stress.

Whereas the issues of resin toxicity and inadvertant etching of the dentine still provoke controversy, it is reasonable to assume, based on the amount of concern demonstrated in the literature over the years, that caution to prevent problems from these two areas is wise, Mjor, (1982). This can be achieved simply by carefully lining the dentine beforehand. However, where a dentine-bonding agent is used instead of a lining there is some evidence to suggest that some reversible pulpal reaction may occur, Franquin et al., (1986). The reaction appears to reverse with time.

Polymerisation contraction occurs with all composite resins and leaves a gap in most cases between the tooth and the restoration. This allows microleakage, and the resultant fluid flows and osmotic pressures brought to bear on the pulp cause pain and sensitivity. Where the resin does not break its bond with the enamel, the opposing enamel margins can be pulled towards one another. This flexes the dentinal tubules and causes pain. In extreme cases cusps can be caused to fracture away from the rest of the tooth.

Deflection of the resin under occlusal stress also allows the tooth to flex, again causing pulpal pain, Suzuki et al., (1985). This can lead to crack

propagation through the dentine and result in catastrophic failure of that part of the tooth. The pain is attributed to changes in hydraulic pressure within the odontoblasts. Johnson et al., (1986), reported greater sensitivity to biting pressure with composite than amalgam or sound tooth. The composite-restored teeth exhibited less sensitivity to temperature change than amalgam.

#### 4. SUMMARY

The rate of change in restorative resin technology, particularly the posterior resins, can be measured by the wide variety and rapidly changing number of available products. It can also be measured by the vast amounts of publications on the subject.

Resin technology has seen changes in the following areas:

The basic resin matrix;

The use of lower viscosity monomers to modify setting and handling;

Changes in filler particle size, shape, and type;

Advances towards radiopaque restoratives;

Improvements in the coupling system between filler and matrix;

The use of photo-activated curing;

Cavity design;

Bonding to the tooth, initially to enamel and now also to dentine;

Improved finishing techniques and better surfaces and aesthetics;

Larger shade ranges and better colour stability;

Improved wear characteristics and form stability;

Reduced water sorption and solubility;

Much greater physical and mechanical properties.

A number of these areas still need improvement, particularly hydrolytic degradation, wear, and curing conversion rate. The interest lies in the amount of controversy on ways to attain improvements.

Areas which appear to generate a lot of comment are the clinical performance versus the in vitro predictions, the production of a system with no polymerisation shrinkage and gap formation, better adhesion of resin to tooth structure, particularly dentine. The latter area has even seen the use of laser technology in the quest to reach better adhesion.

Perhaps the greatest area of concern is the rate at which materials with no long-term clinical trials are being forced onto the commercial market with little consensus amongst the researchers on a standard system to assess the possible performance of the product.

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## APPENDIX B.

## LIST OF RESINS AND MANUFACTURERS, USED IN THIS PROJECT.

Adaptic II	Johnson & Johnson Dental Products Company. 20 Lake Drive, CN 7060, East Windsor, N.J. 08520
Distalite	Johnson & Johnson Dental Products Company. 20 Lake Drive, CN 7060, East Windsor, N.J. 08520
Estilux Posterior	Kulzer & Co GmbH Bereich Dental Postfach 1320 D-6382 Friedrichsdorf 1 Tel. 061 72/7 32-0 Tx. 415 863 kulz d
Fulfil.	L. D. Caulk Div., Dentsply Int. Inc., Milford, Delaware, 19963-0359 U.S.A.
Heliomolar	Vivadent Schaan Liechtenstein
Herculite Syringeable. Shades; Universal Light Yellow Grey	Kerr, Romulus, Michigan, 48174, U.S.A.
LC 1000	Southern Dental Industries Melbourne Australia.
Occlusin Shades: XL, S, DY & LG	Imperial Chemical Industries PLC Pharmaceuticals Division Alderley House, Alderley Park Macclesfield, England

P-30	Dental Products Division/3M
Shades:	225-1S-09 3M Center,
XL, U, Y& G	St. Paul, MN 55144. U.S.A.
Sinterfil	Teledyne Getz
	Buffalo
	New York
	U.S.A
Visio Molar	ESPE.
Shades:	Fabrik Pharmazcutischer
Standard	Paraparetc
Grey	G.M.B.H.
Brown	D-8031 Seefeld/Oberbay.
Light	

APPENDIX C.  
CASE REPORT  
POSTERIOR RESINS

The purpose of including this appendix is to high-light the need to adhere to good conservative principles when using these materials. When used inappropriately the consequences can be disastrous as seen in this case. The practitioner used materials in situations that were far behind their designed capabilities, he did not respect good technique, ( in fact we know from the patient's history that rubber dam was not used ), and he had not diagnosed the extent of the problems in this mouth correctly.

**PERSONAL DETAILS**

Name: \*\*\*\*\*.  
Case note: 119627  
Date of birth: 22/11/24.

**CHIEF COMPLAINT:**

This patient has had extensive rehabilitation with posterior composite resin. The subsequent secondary caries and pulpal involvement have created occlusal, functional and aesthetic problems of enormous proportions.

**REFERRAL:**

Primary Care Unit , Adelaide Dental Hospital

**MEDICAL HISTORY:**

Nil of note.

**PAST DENTAL HISTORY:**

1. A number of extractions
2. Inadequately treated root canals
3. Severe bruxism, with headaches left untreated.
4. Full gold crowns
5. Extensive posterior resins ( See figures D1 and D2 )

**Figure D 1**

Photograph of the patient's lower right second premolar. This tooth had been restored with a composite resin MOD restoration. The gingival retraction cord was placed to displace the hyperplastic gingiva from the cavity under the mesial aspect of the filling to enable a clearer photograph of the poor margin and the carious lesion.

**Figure D 2**

This photograph of the patient's upper arch shows the extent of the use of resin restorations. A number of the teeth have full crown composite resins several of which have now been endodontically treated and are to be replaced with conventional crowns. These restorations are all less than two years old.



**DIAGNOSIS:**

1. Extensive breakdown of inappropriate restorative material.
2. A highly nervous patient, who bruxes very hard.
3. Recurrent caries under most restorations with pulpal involvement in some teeth.

**TREATMENT RENDERED:****Preventive:**

The first step in this restorative program for this lady was to raise the level of her awareness for personal oral hygiene. The patient was referred to the hospital hygienists following an initial scale and clean by the operator. The referral required O.H.I. and continued maintenance of the gingival plaque levels.

**Simple Conservative Work:**

- 45 Canal obliterated with AH26 & G.P.
- 17 MOD Amalgam 2 T.M.S. Minim Pins
- 16 MODBL Amalgam 2 T.M.S. Minim Pins
- 13 DMP Ketac-bond/Silux.
- 11 DPIn. Ketac-bond/Silux. 1 T.M.S. Minim Pin
- 26 MODBL Amalgam 3 Paraposts ( yellow )
- 22 MDPLa Ketac-bond/Silux.
- 21 DPInLa Ketac-bond/Silux.
- 37 MODL Amalgam post/ core crown.
- 34 MO Amalgam
- 33 DB Amalgam
- 43 B Ketac-fil
- 45 MOL Amalgam 2 T.M.S. Minim Pins
- 48 MODB Amalgam.

**Removable Prosthetic Work:**

Clear acrylic splint

**Complex Conservative work:**

No complex treatment plan has been discussed at this stage, although one will be required if the patient can show an improvement in oral hygiene control.

**SEQUENCE OF TREATMENT:**

Stabilisation & Evaluation Phase:

Exam.

Study models and photographs.

Full mouth survey and O.P.G.

O.H.I.

Simple Conservative Restorations and cores.

Minor oral surgery, removal of the 44 root fragment.

Review.

**PROGNOSIS.**

The prognosis for the success of any treatment plan for this patient is heavily dependant on her ability to maintain her oral hygiene at an acceptable level, and to control the severe bruxism.

APPENDIX D.  
AUSTRALIAN STANDARD  
1278 - 1982  
RESIN BASED DENTAL RESTORATIVE MATERIALS.  
METHOD FOR DETERMINING DEPTH OF CURE.

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Standards Association of Australia

Standards House,

80 Arthur Street,

North Sydney, N.S.W.



**C1 SCOPE.** This Appendix sets out the method for determining the depth of cure of Type II resin-based dental restorative materials, after activation of the material with the recommended external energy source for the recommended time.

**C2 PRINCIPLE.** The distance between the top surface of a cylindrical specimen which has been irradiated, and the region in the material at which little polymerization has occurred is measured by a micrometer to give the depth of cure.

**C3 APPARATUS.** The following apparatus is required:

- (a) An open-ended, split stainless steel mould for the preparation of a cylindrical specimen 8.0 mm high and 4.0 mm in diameter.
- (b) Two microscope slides, each of sufficient area to cover one end of the mould.
- (c) Clear, UV-transparent film, e.g. Cellophane.
- (d) The external energy source recommended by the manufacturer.
- (e) A small clamp.
- (f) A micrometer.

**C4 PREPARATION OF SPECIMEN.** Place the mould on a strip of the UV-transparent film and fill the mould with the material, prepared in accordance with the manufacturer's instructions, taking care to exclude air bubbles. Slightly overfill the mould. Press the mould and strip of film between the glass slides to exude excess material. Remove the microscope slide covering the strip of film and gently place the exit window of the external energy source against the strip of film. Irradiate the material for the time period recommended by the manufacturer.

**C5 PROCEDURE.** At  $180 \pm 20$  s after completion of exposure, remove the specimen from the mould and strip of film, and gently remove the uncured material by means of a plastics spatula. Measure the maximum thickness of the cured material with a micrometer to the nearest 0.01 mm.

**C6 REPORT.** The depth of cure of the specimen shall be reported to the nearest 0.1 mm.

**Depth of Cure: verification of AS 1278-1982**

Material: Herculite Syringeable, Universal shade.

Batch Number: P8506 5 3144 1

Sample size : 10                      Range : 4.7 mm - 4.98 mm

Measurements.	#1	4.72 mm
	#2	4.98 mm
	#3	4.74 mm
	#4	4.70 mm
	#5	4.72 mm
	#6	4.82 mm
	#7	4.84 mm
	#8	4.72 mm
	#9	4.74 mm
	#10	4.92 mm

Mean : 4.79 mm

Standard deviation : 0.1

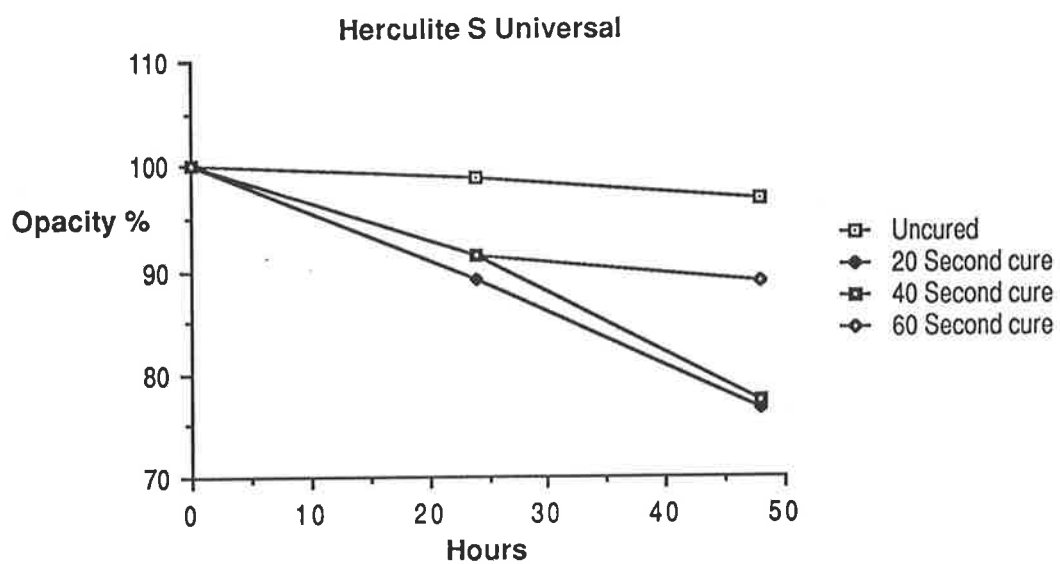
## APPENDIX E. OPACITY TESTS:

## GRAPHIC DISPLAYS OF

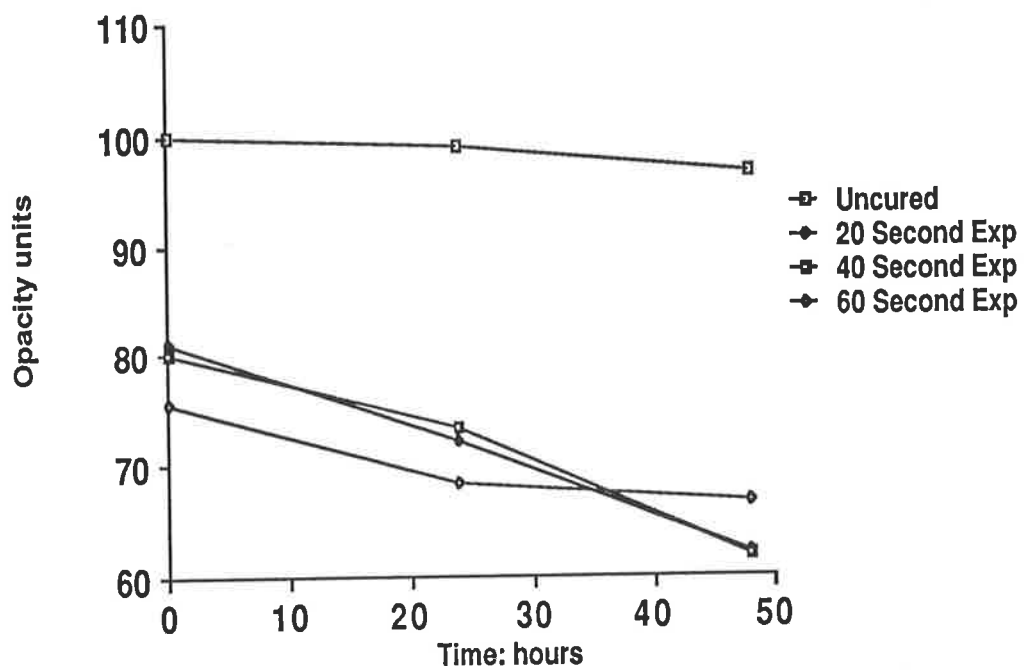
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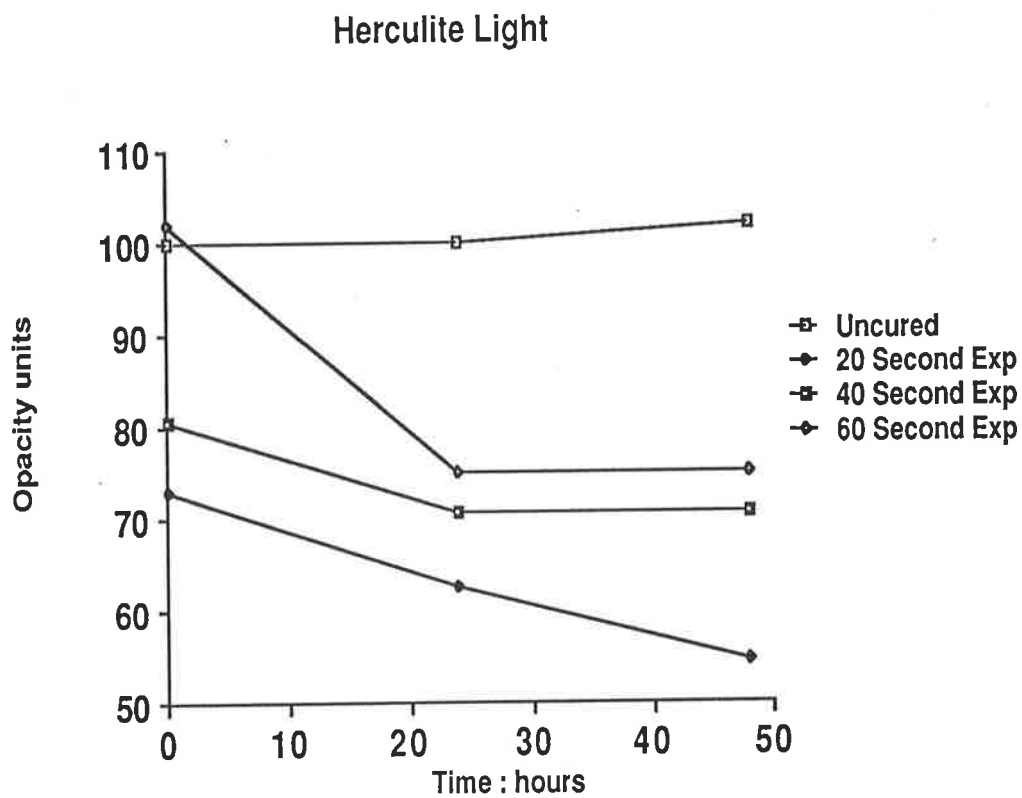
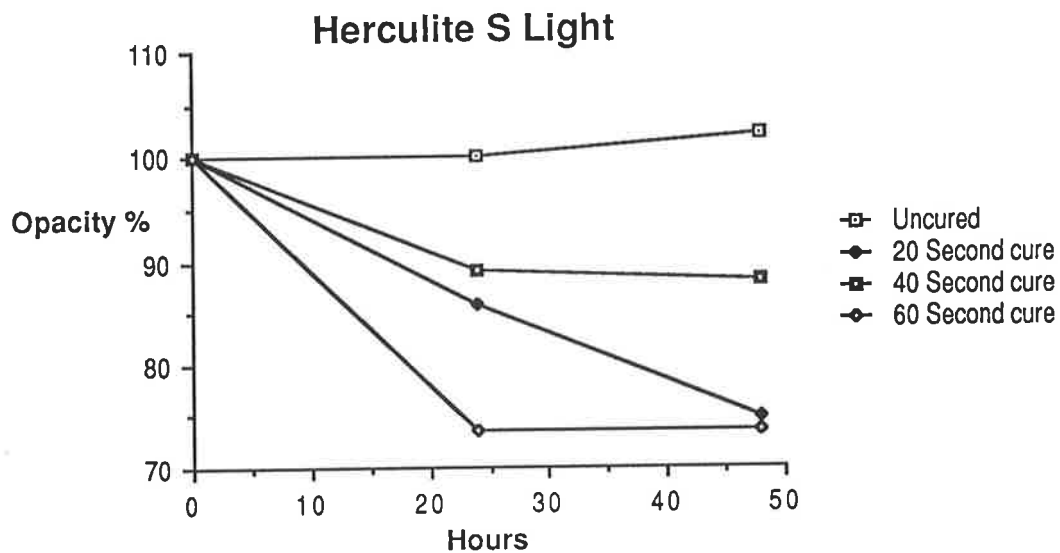
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5.3.2.a - n Graphs for individual resin opacity changes.

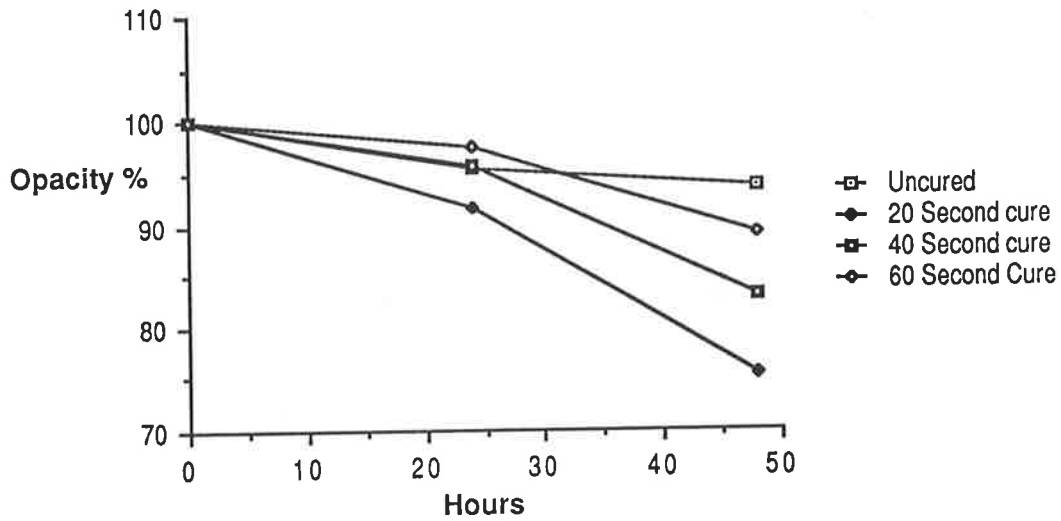


## Herculite Universal

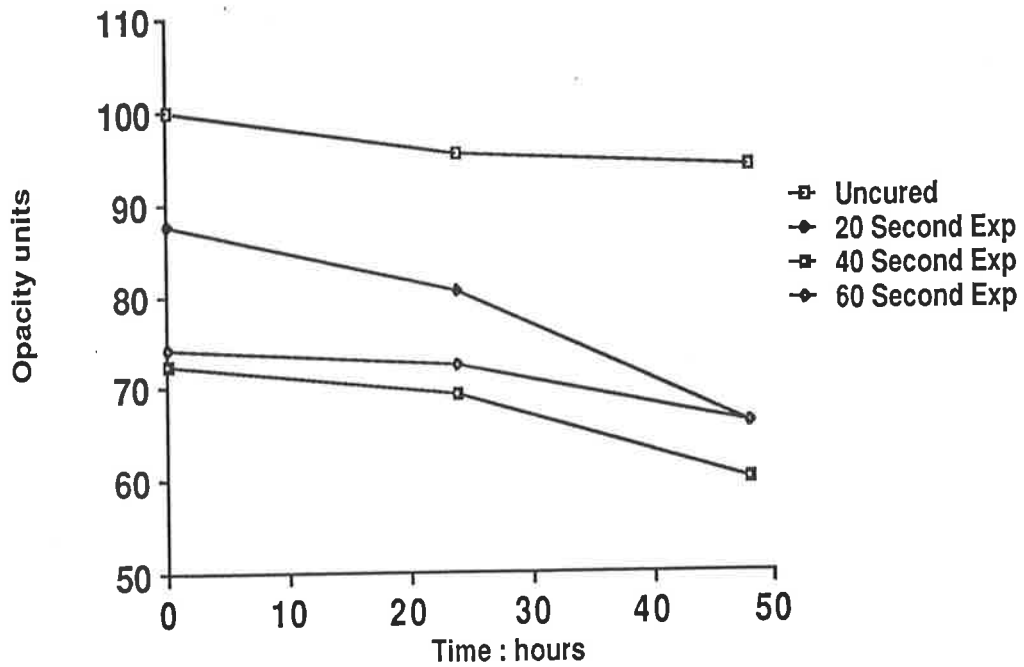




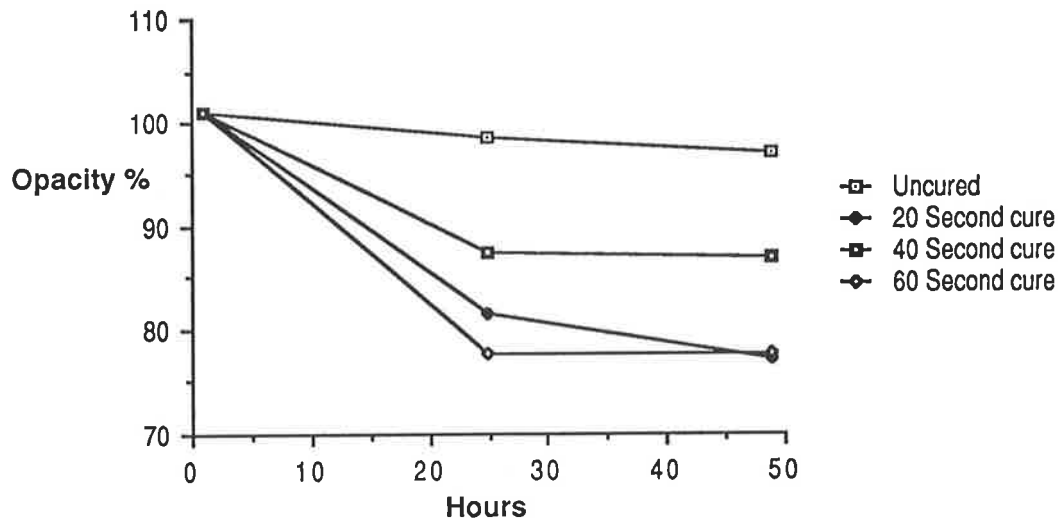
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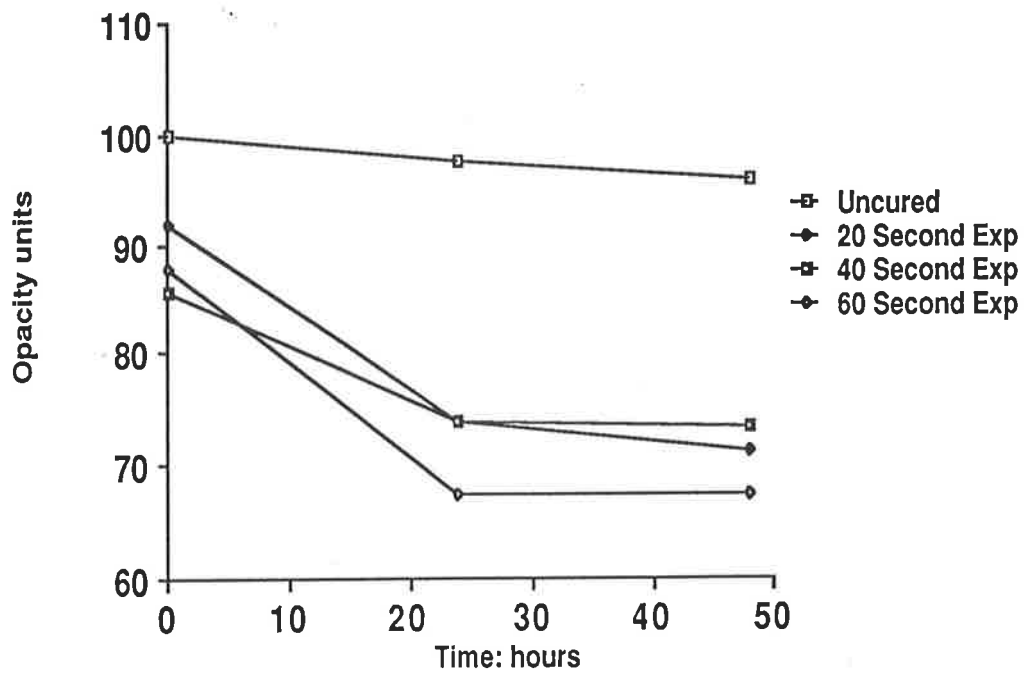
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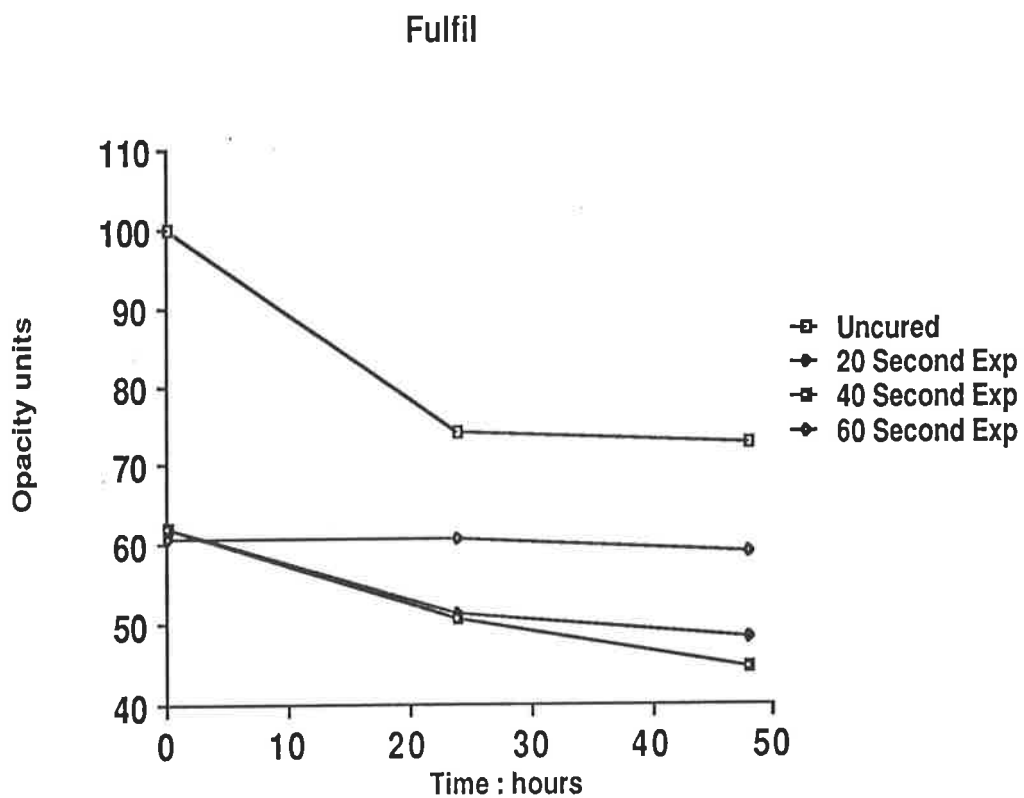
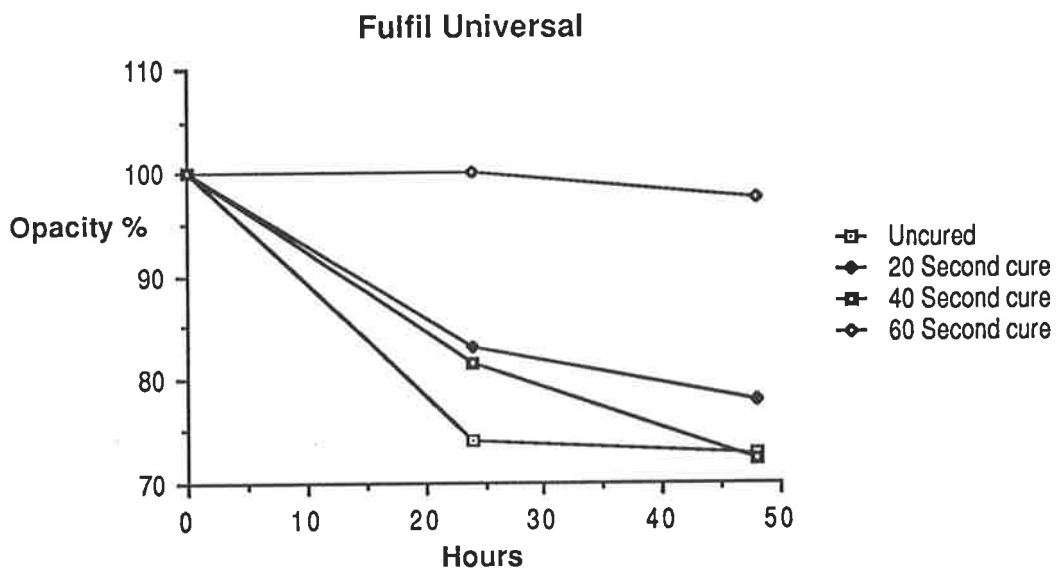


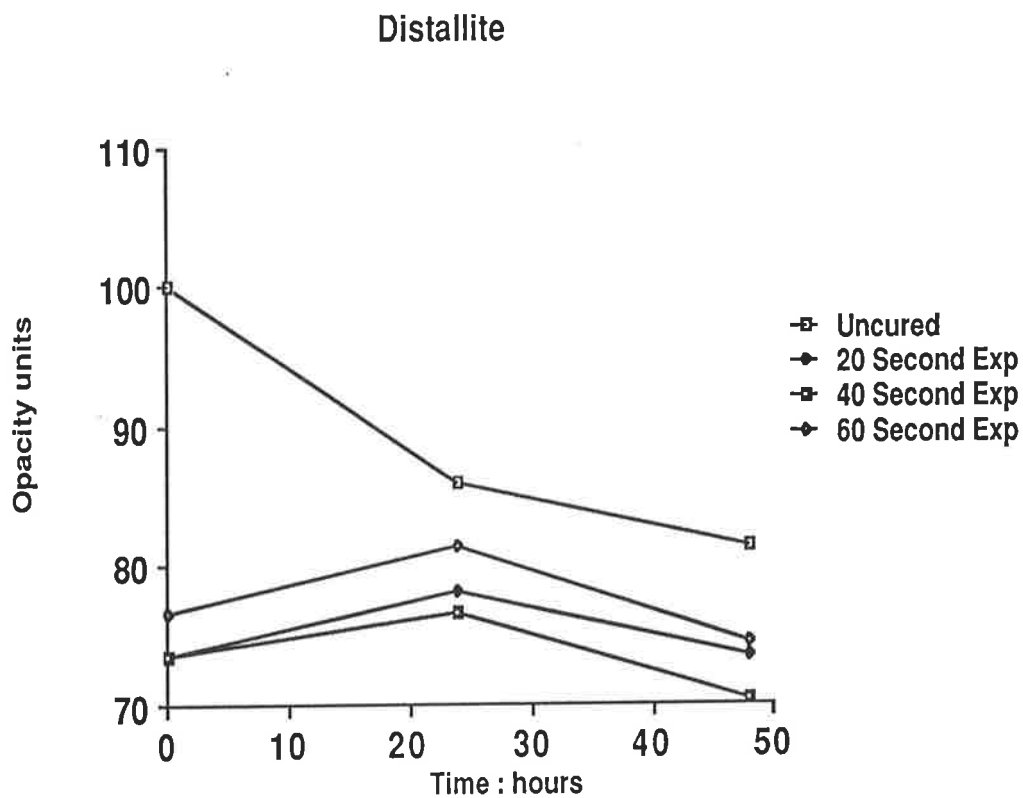
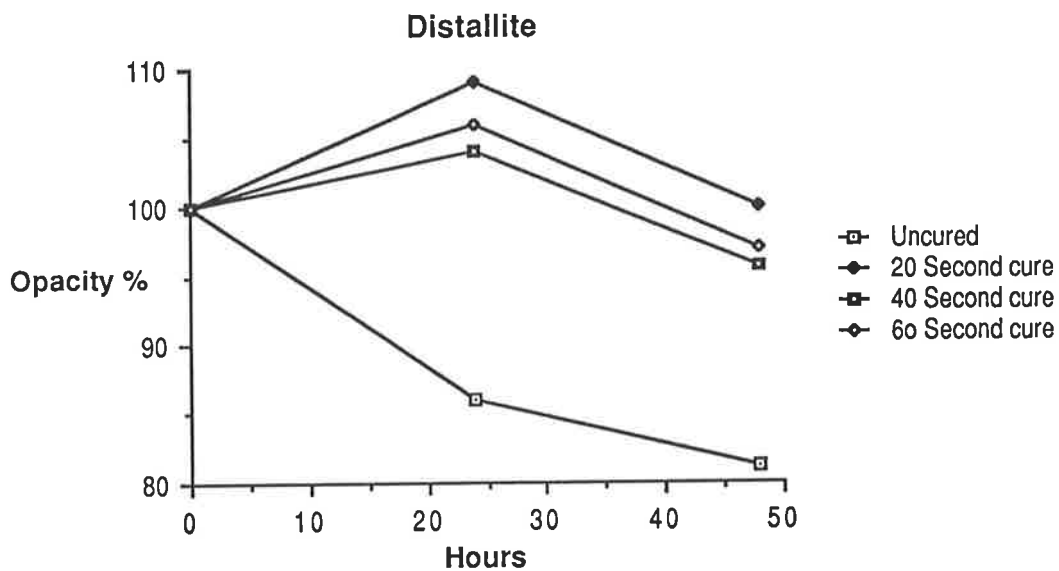
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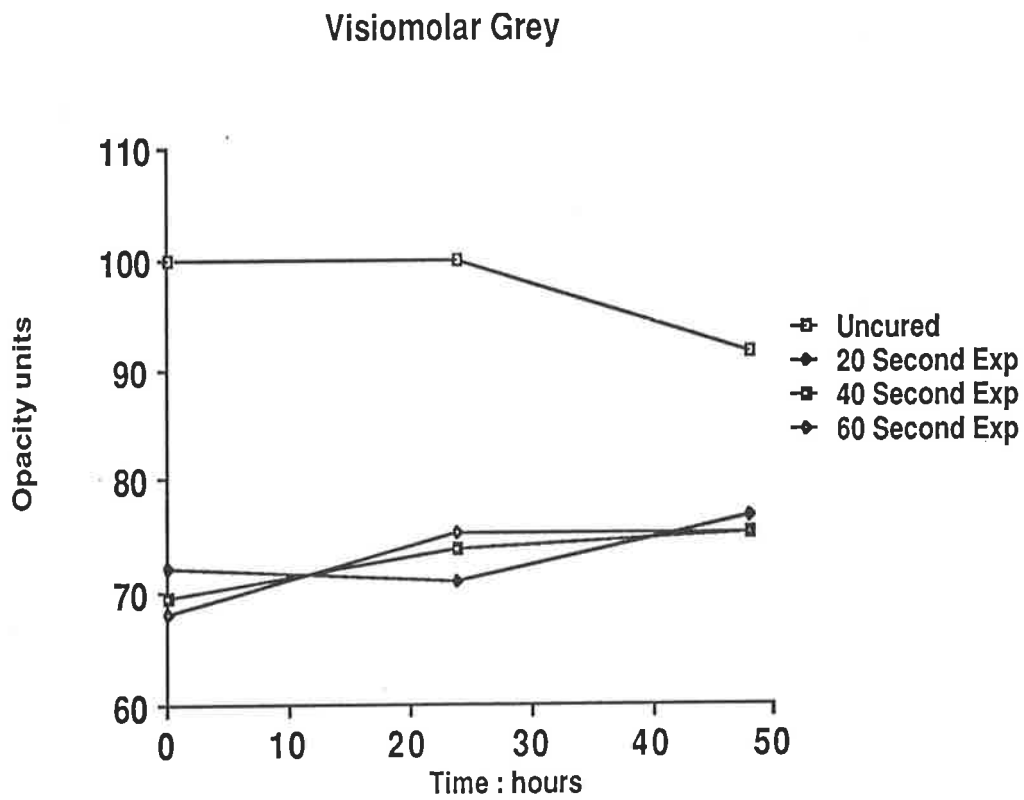
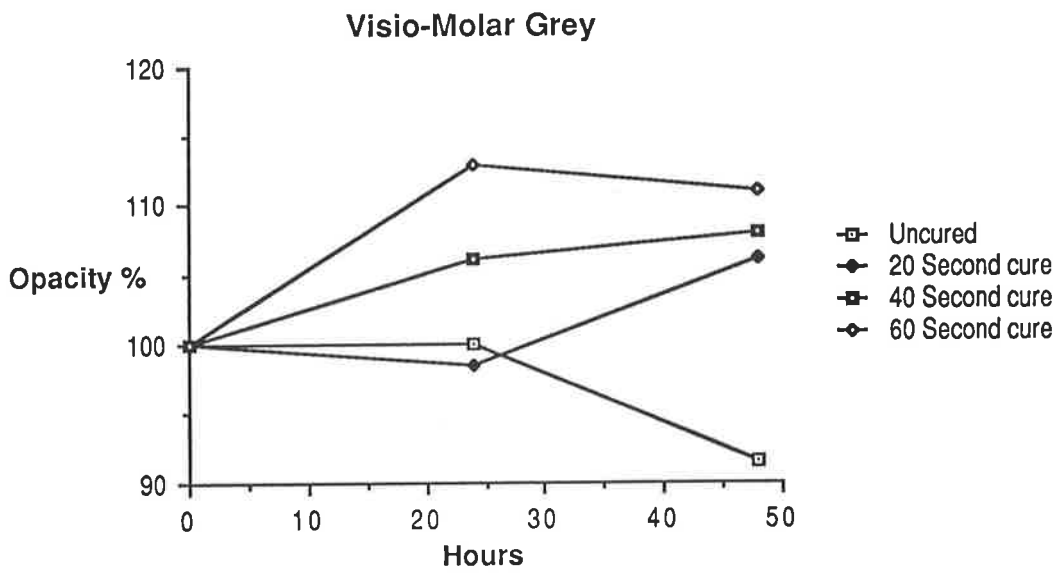


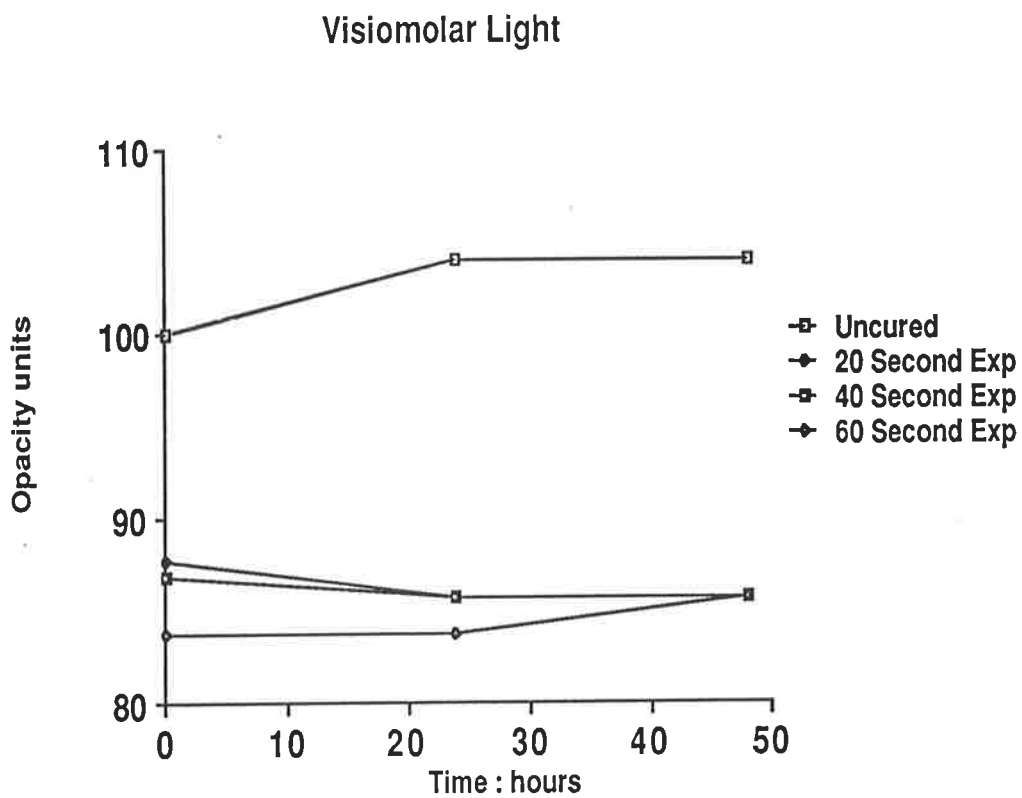
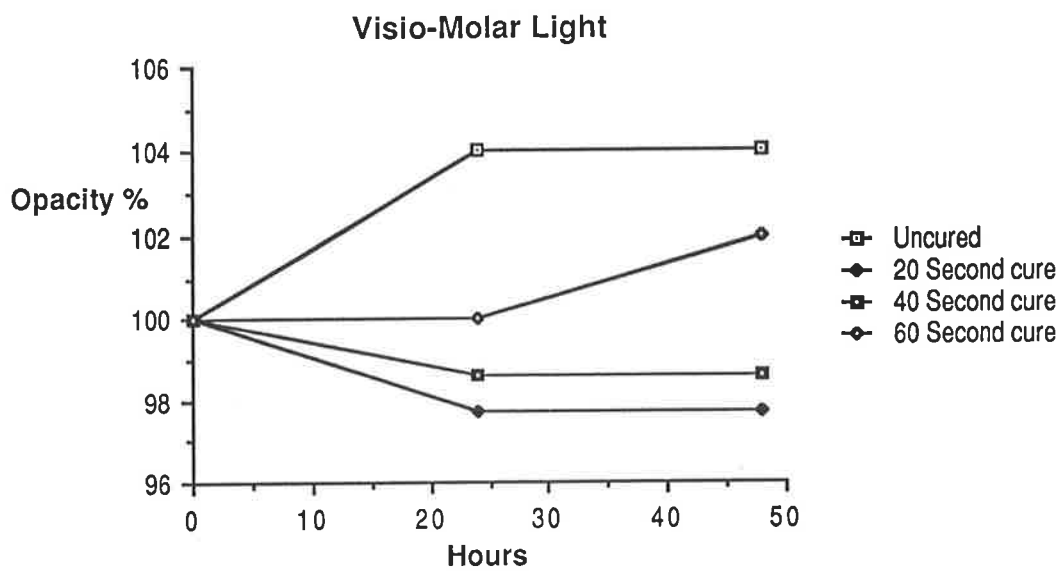
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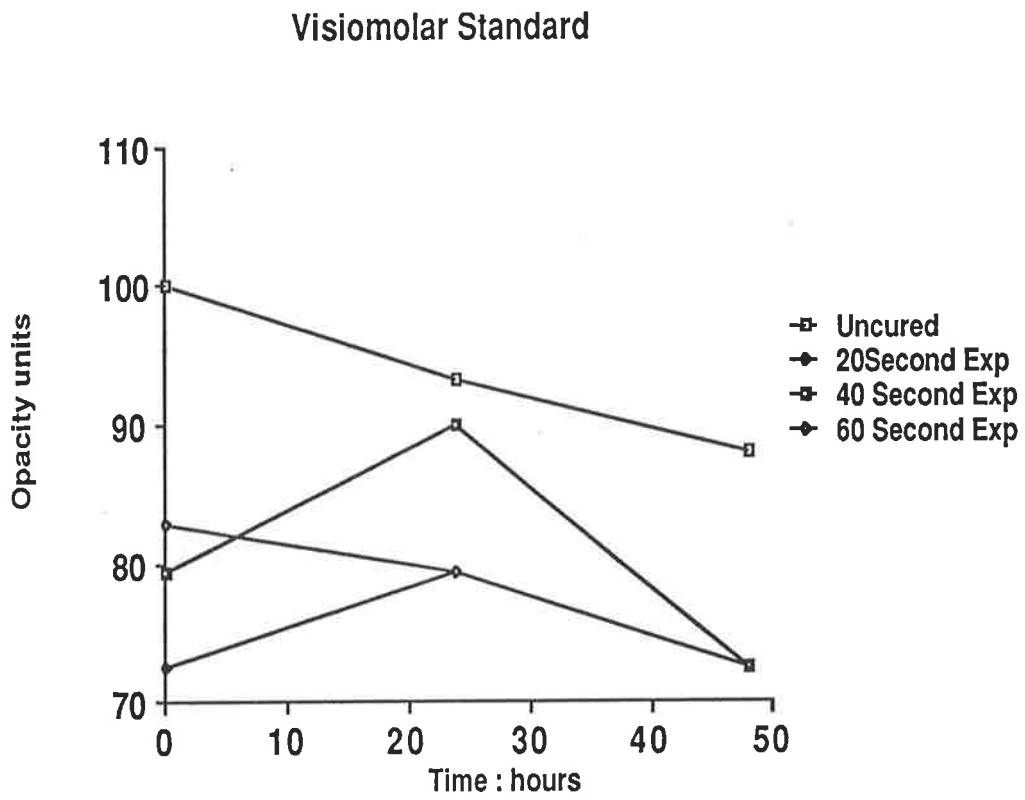
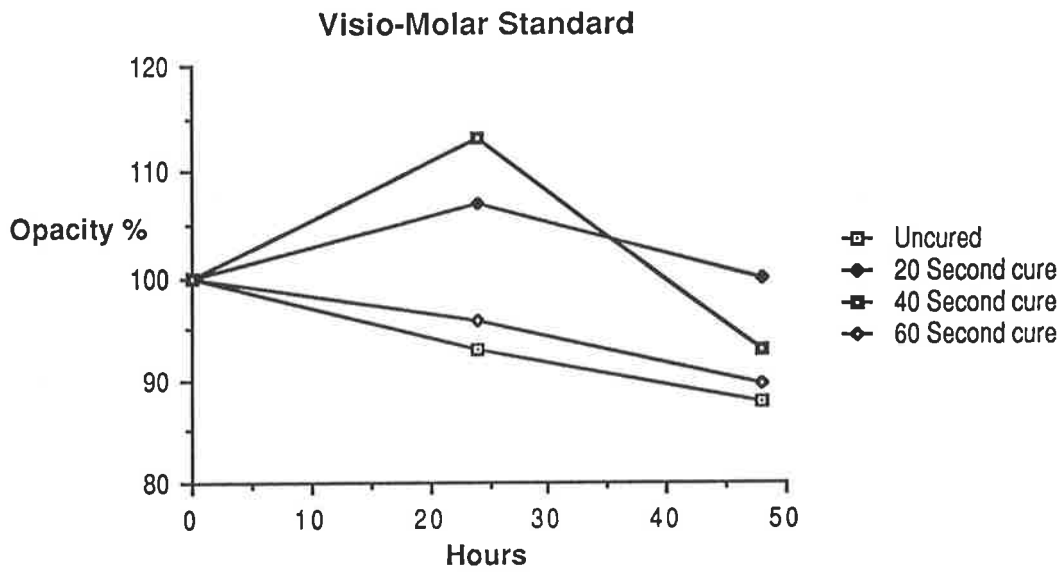


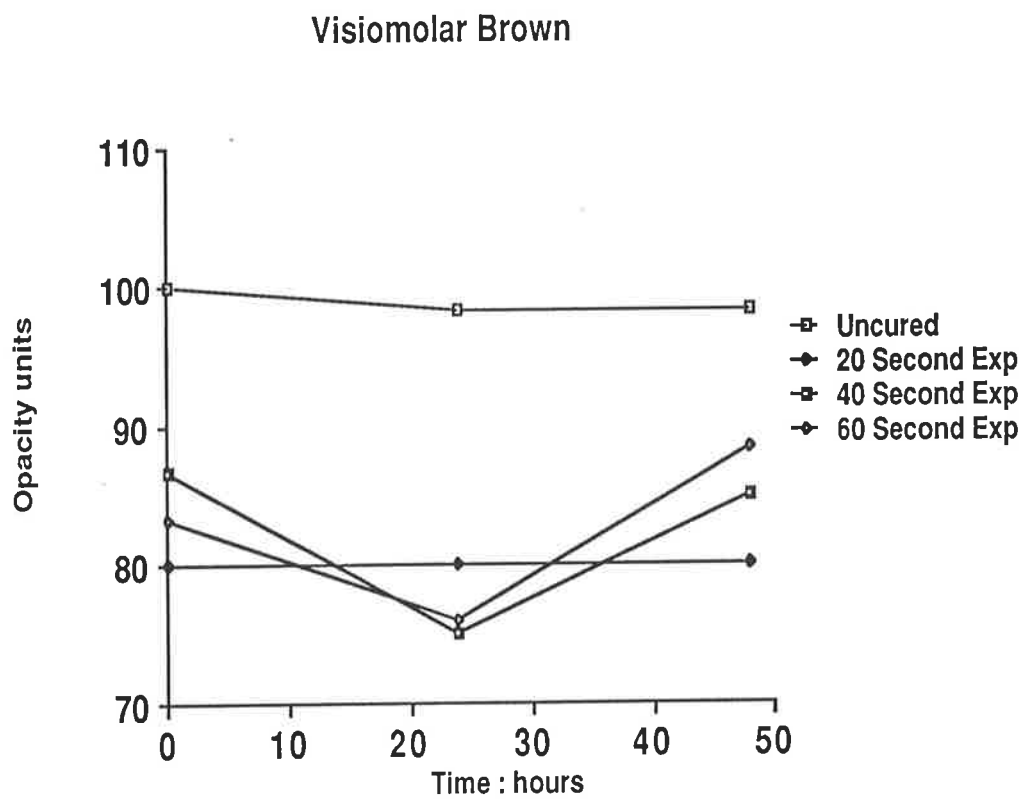
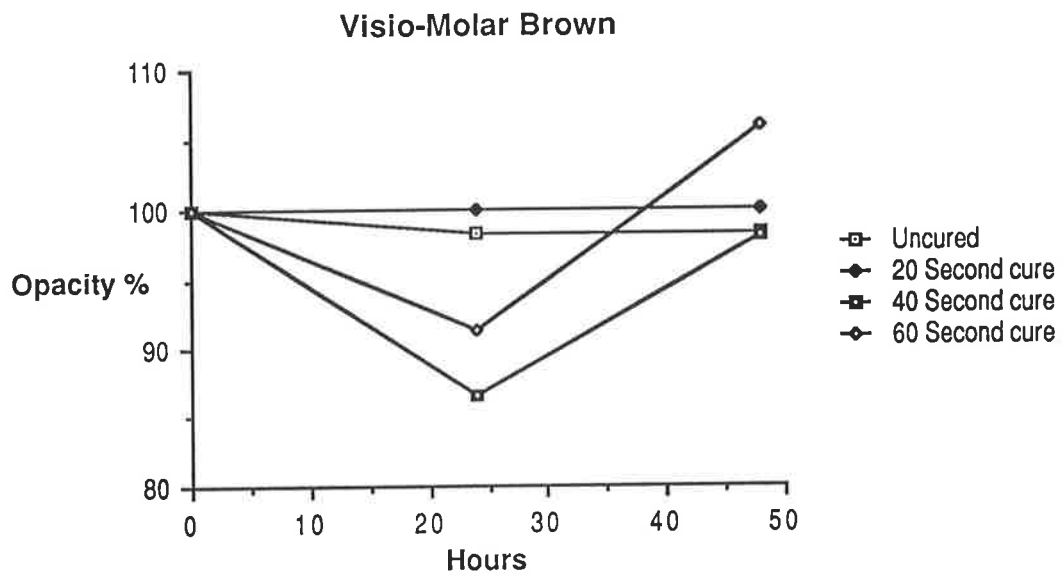




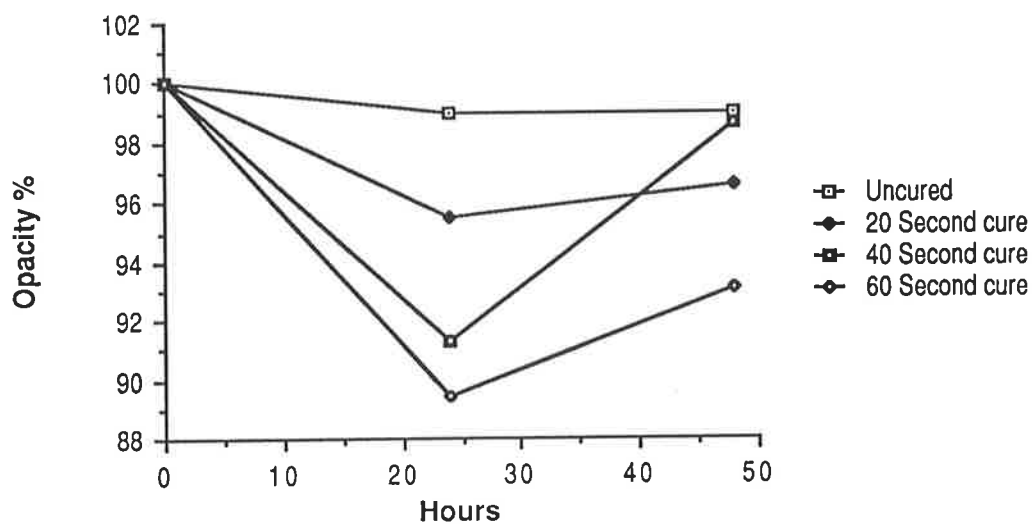




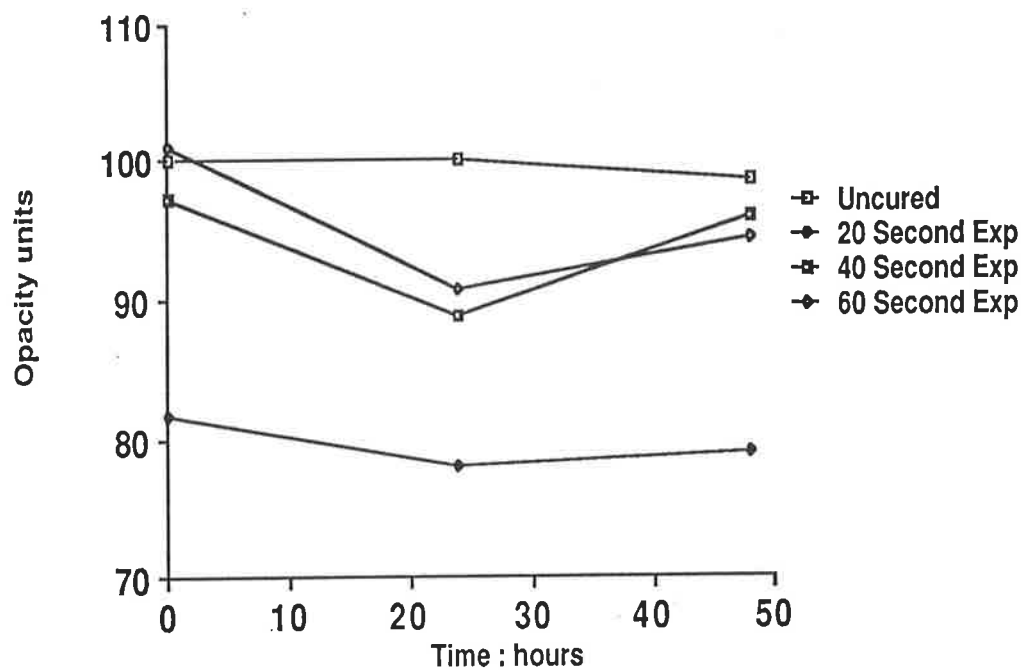




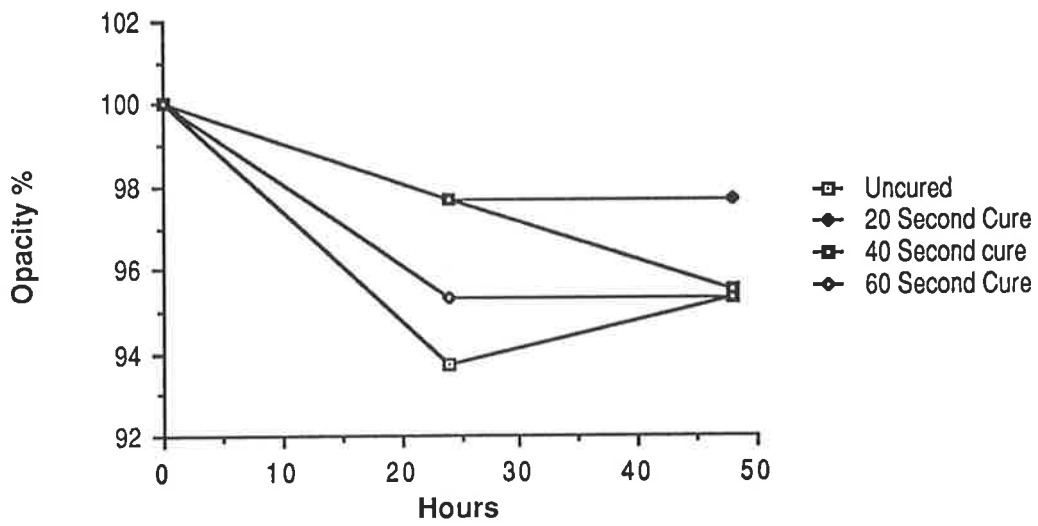
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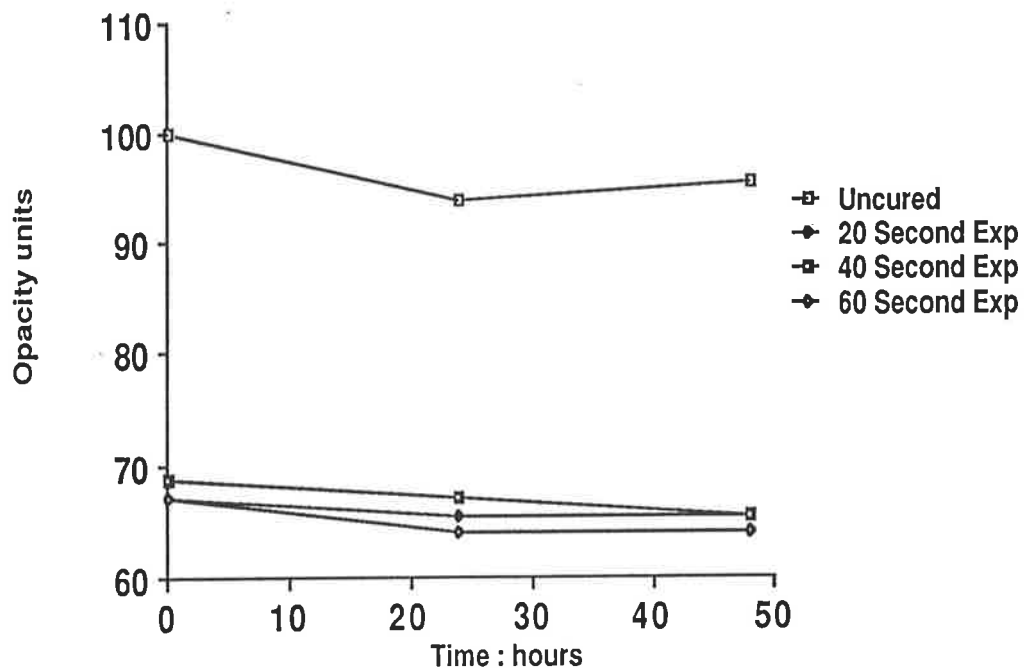
Adaptic II



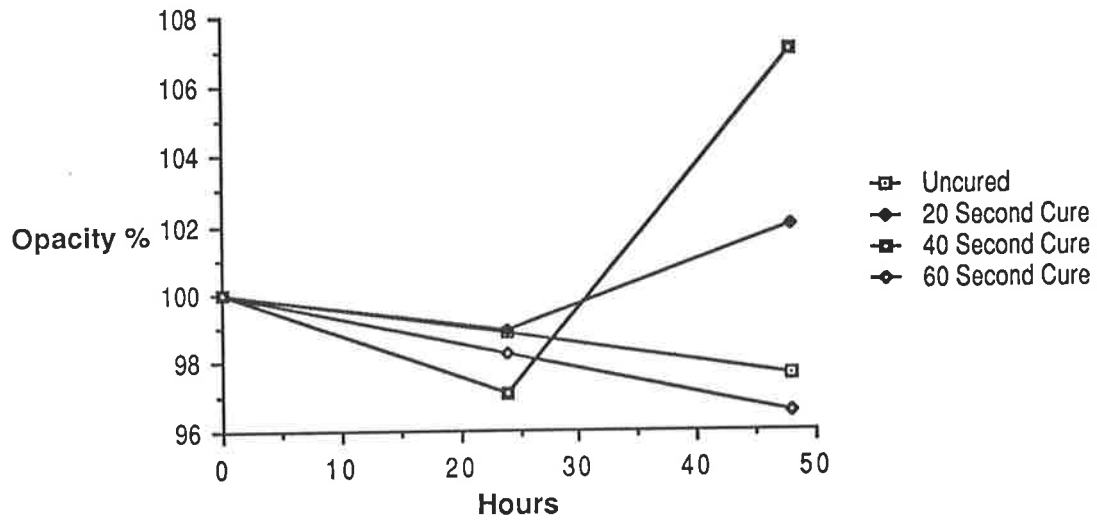
Occlusin XL



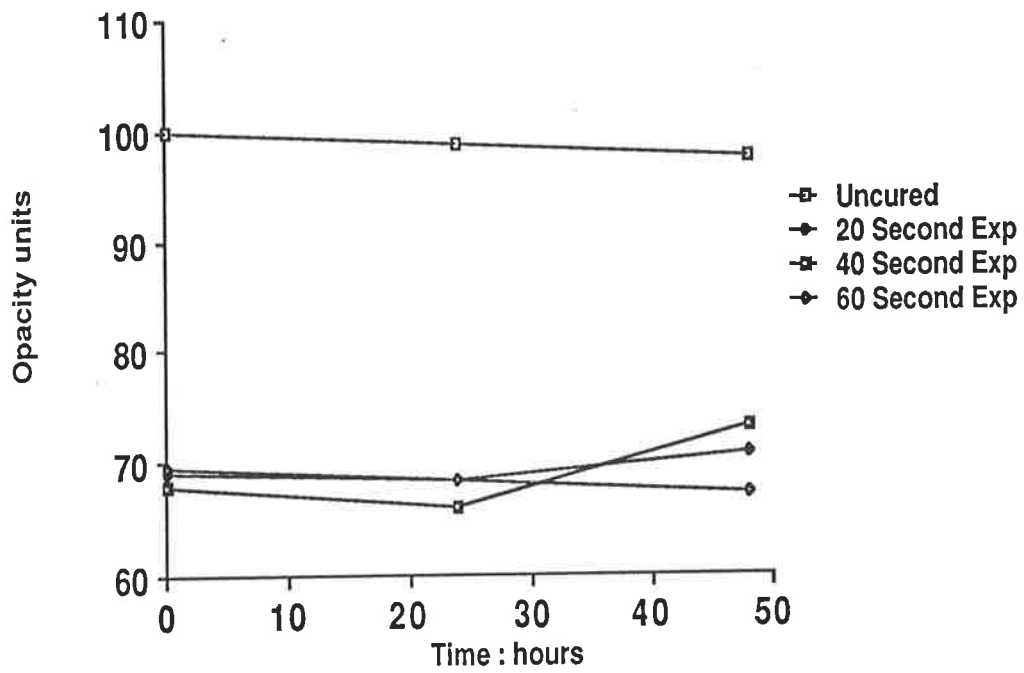
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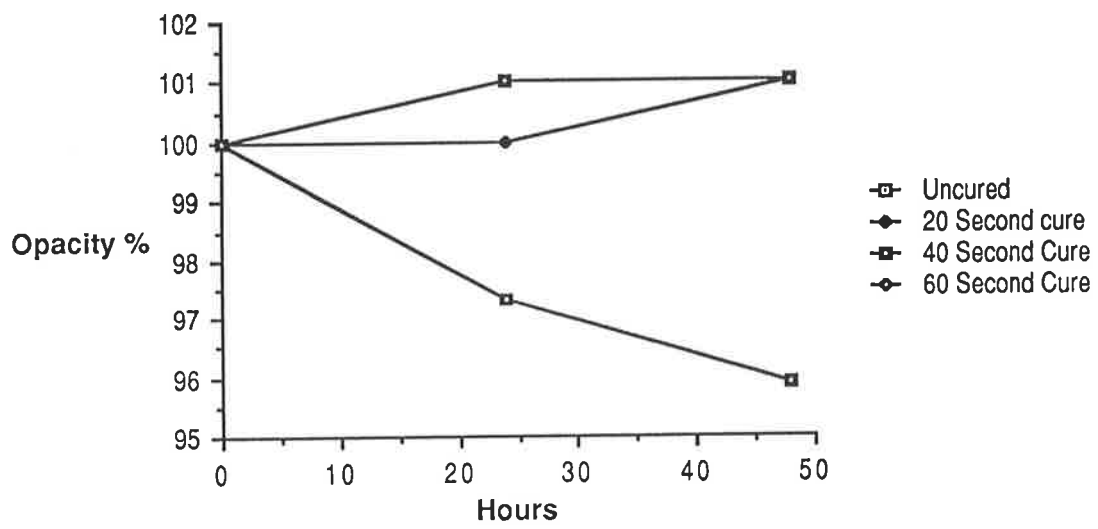
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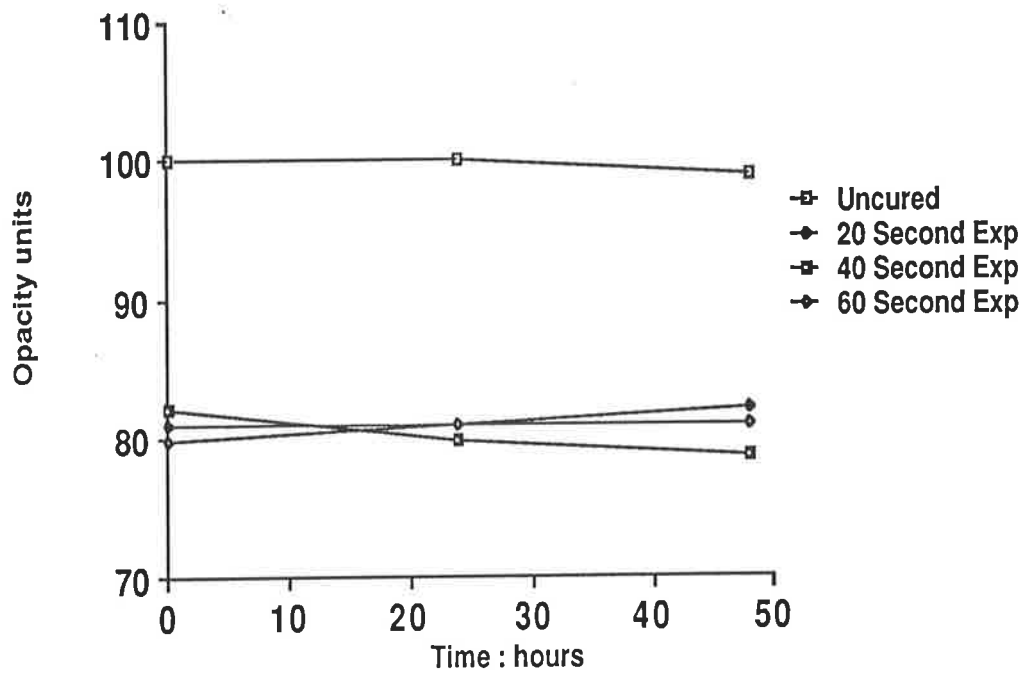
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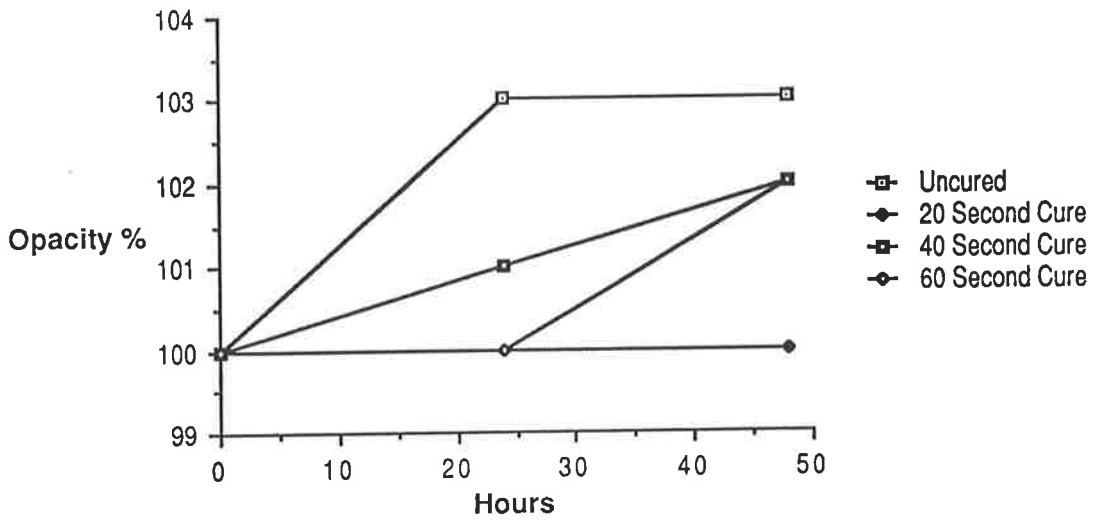
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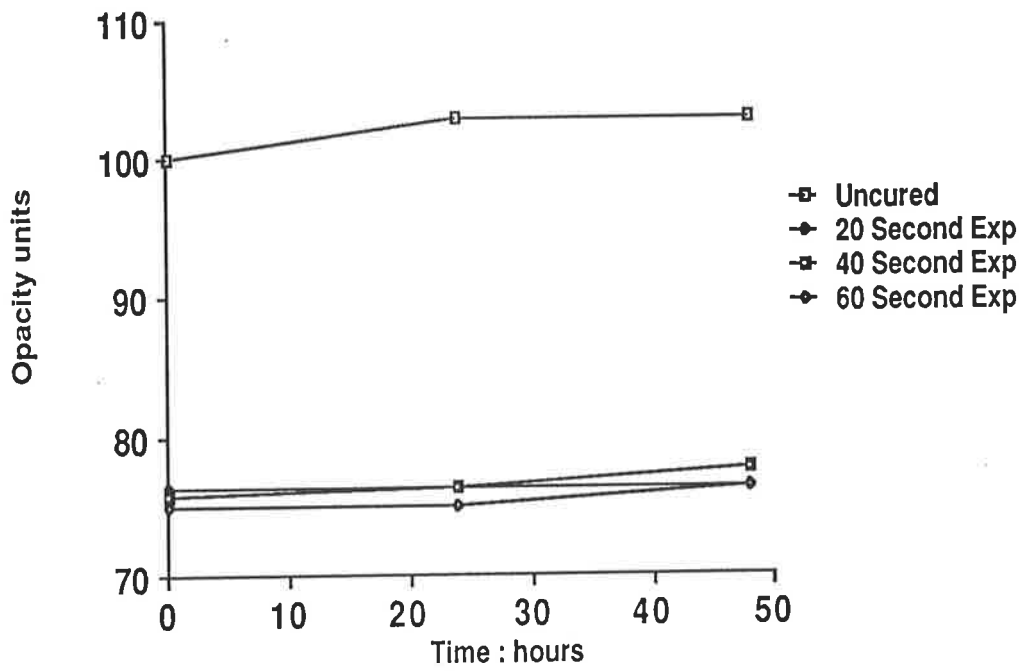
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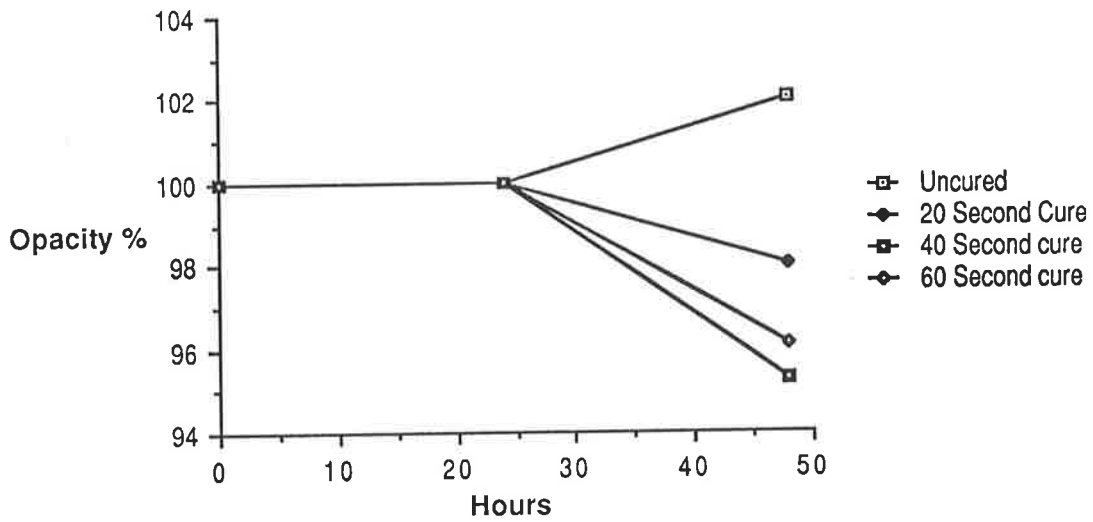
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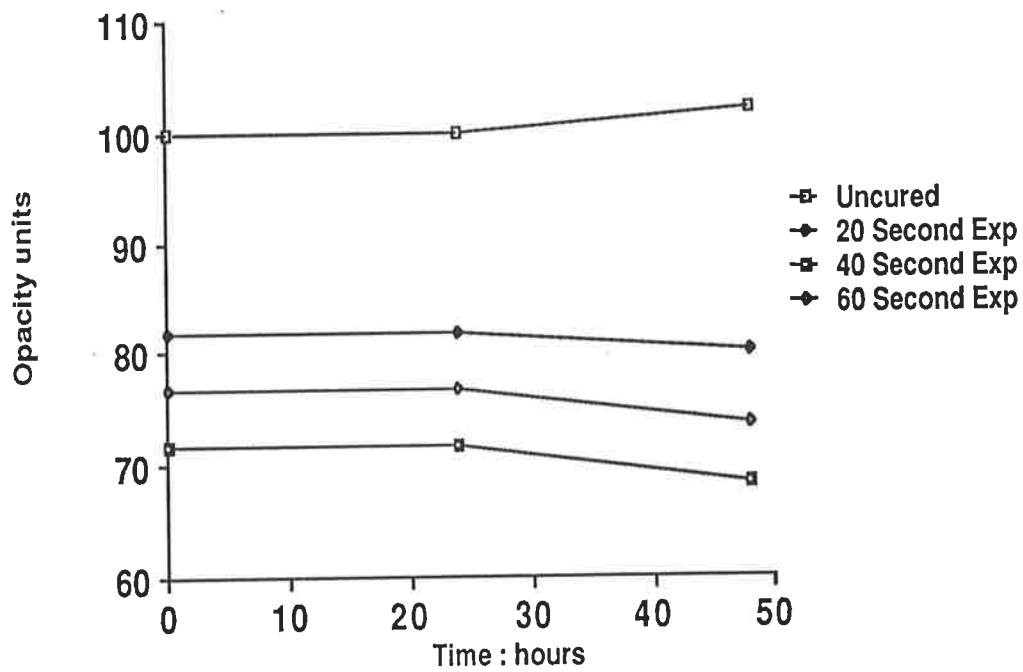
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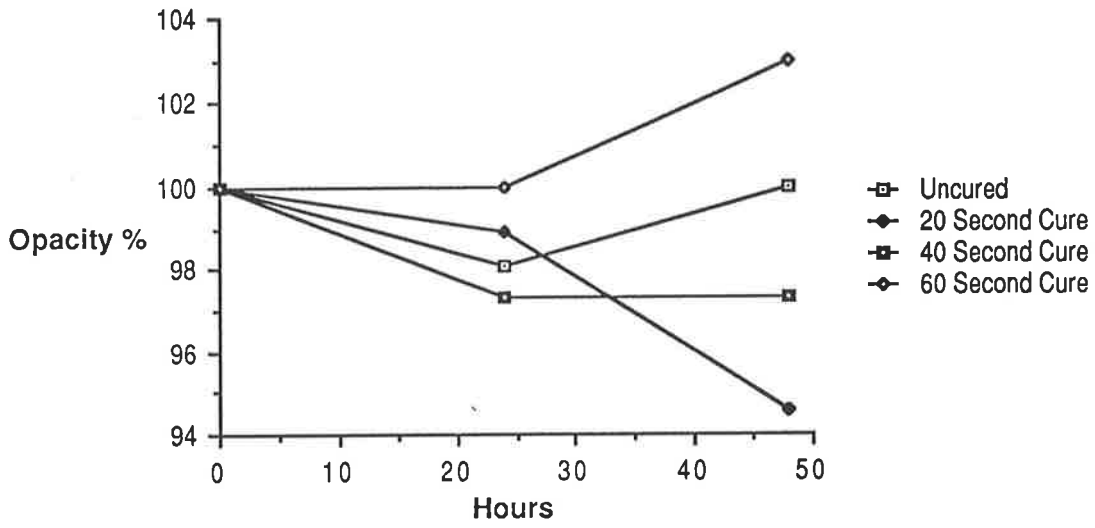
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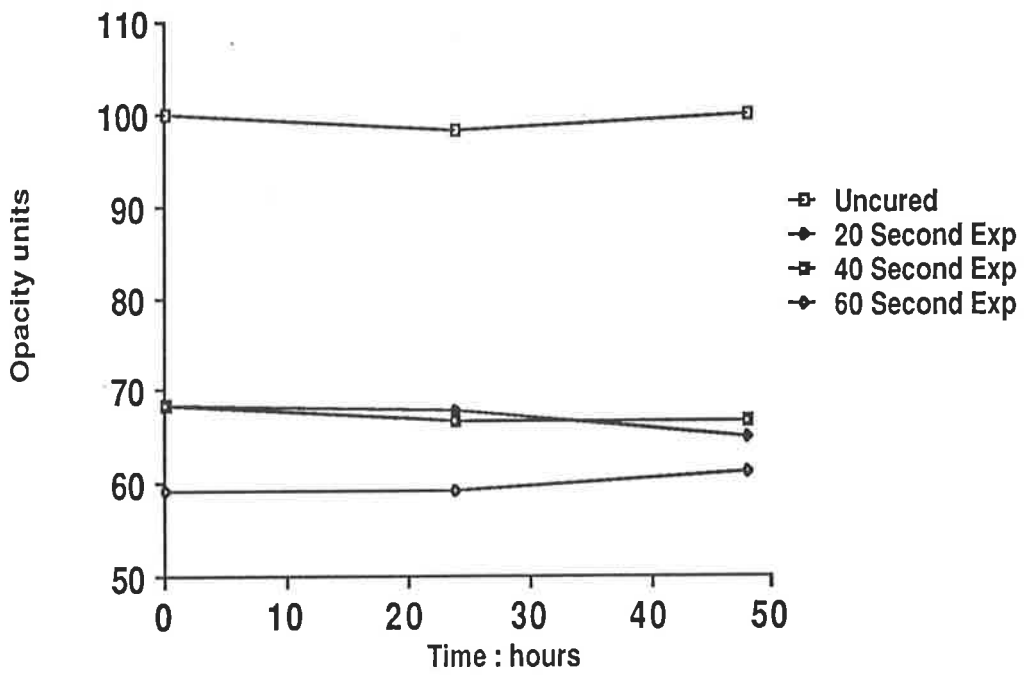
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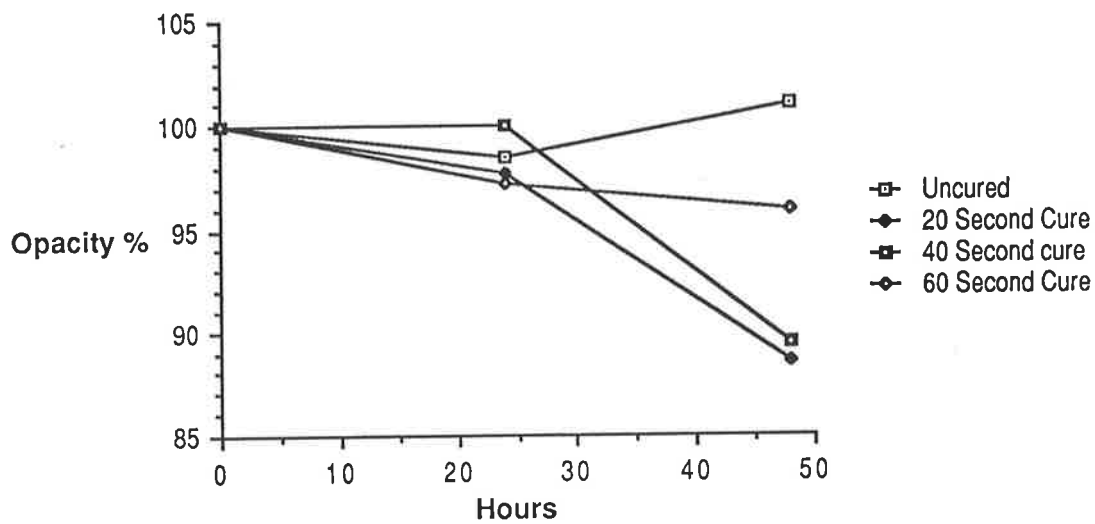
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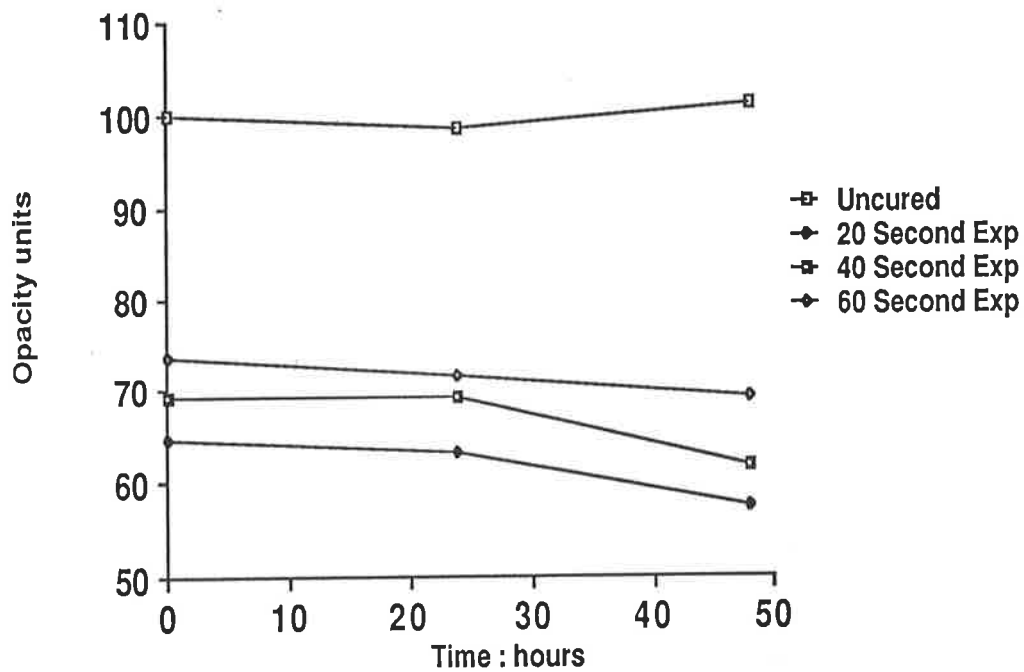
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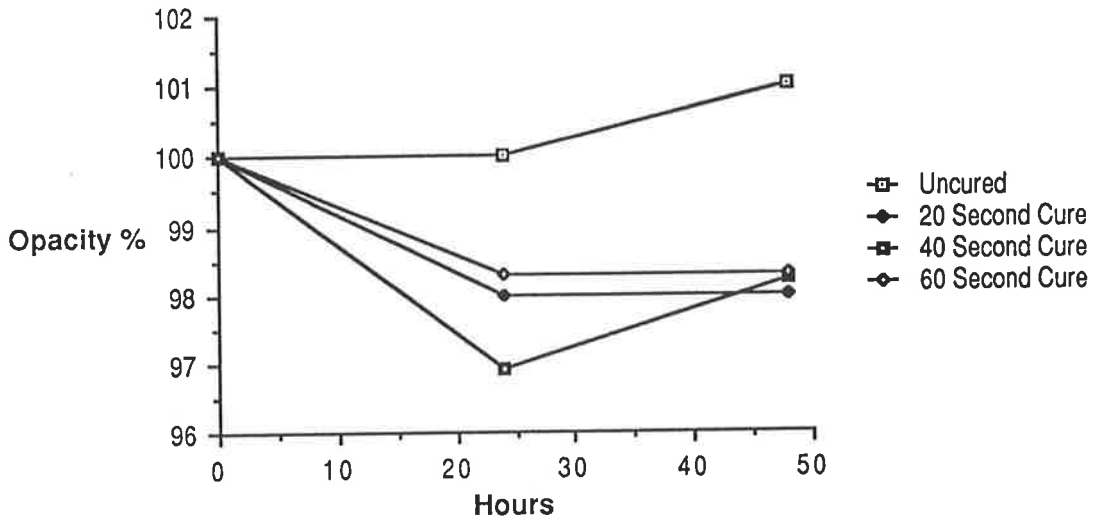
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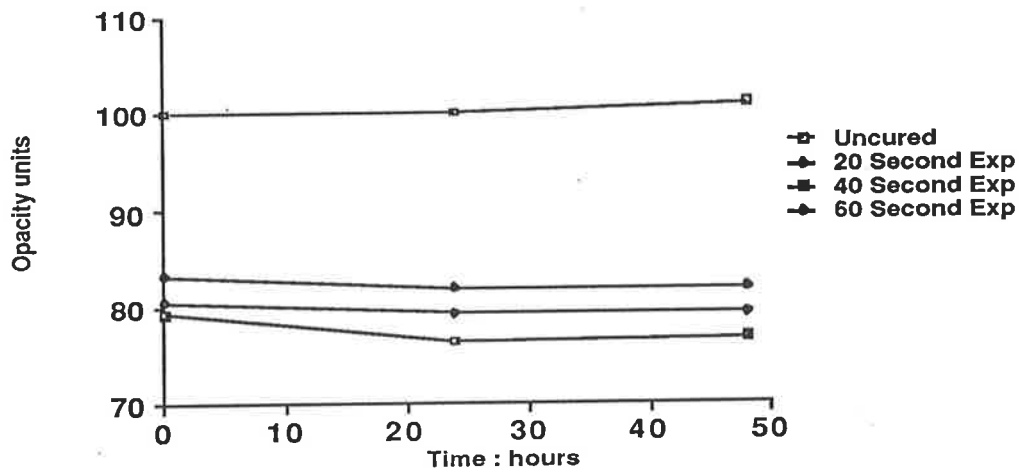
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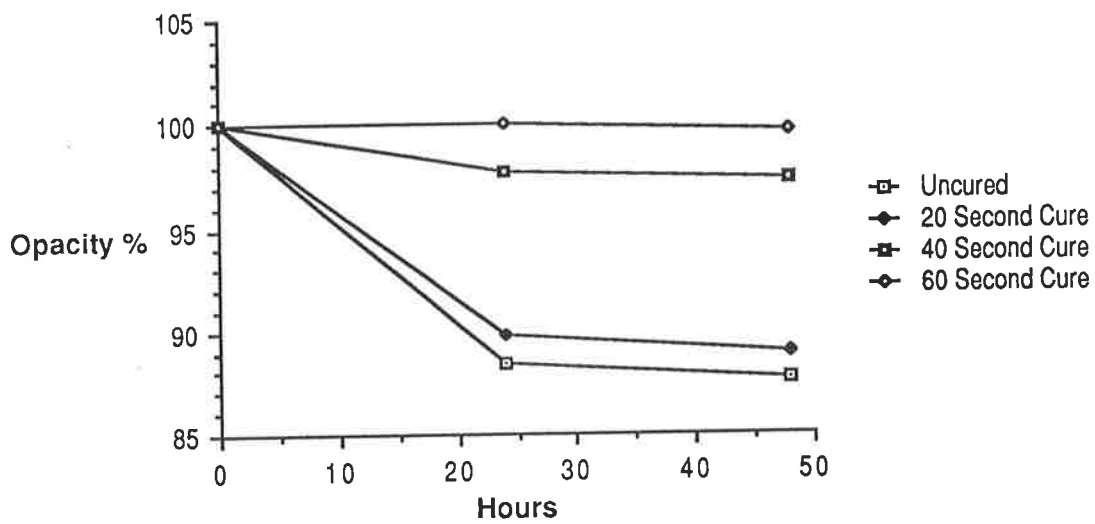
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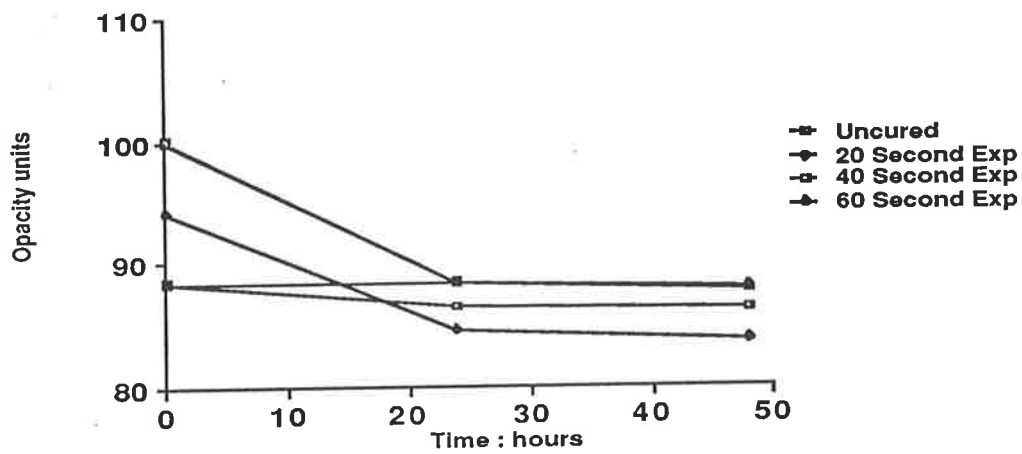
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Estilux Posterior



Estilux Posterior



Specimen Number	Expo-	Times when measured post curing							
		0 Hours		24 Hours		48 Hours			
sure									
		(Secs)							
Herculite	246	0	0.245	100	0.239	97.6	0.235	96	
Yellow	247	20	0.225	91.8	0.181	73.9	0.171	71.3	
	248	40	0.21	85.7	0.181	73.9	0.180	73.5	
	249	60	0.215	87.8	0.165	67.3	0.165	67.3	
Herculite	239	0	0.28	100	0.28	100	0.275	98.2	
Universal	240	0	0.33	100	0.325	98.5	0.310	93.9	
	241	0	0.29	100	0.285	98.3	0.285	98.3	
	230	20	0.25	100	0.215	86	0.175	70	
	231	20	0.25	100	0.225	90	0.202	80.8	
	232	20	0.23	100	0.21	91.3	0.182	79.1	
	233	40	0.23	100	0.195	84.8	0.183	79.6	
	234	40	0.24	100	0.230	95.8	0.192	80	
	235	40	0.25	100	0.235	94	0.182	72.8	
	236	60	0.23	100	0.195	84.8	0.195	84.8	
	237	60	0.2	100	0.21	105	0.195	97.5	
	238	60	0.25	100	0.21	84	0.21	84	
Herculite	242	0	0.24	100	0.24	100	0.245	102	
Light	243	20	0.175	72.9	0.15	62.5	0.131	54.6	
	244	40	0.193	80.4	0.172	70.8	0.17	70.8	
	245	60	0.245	102	0.18	75	0.18	75	
Herculite	250	0	0.325	100	0.310	95.4	0.305	93.8	
Grey	251	20	0.285	87.7	0.261	80.3	0.215	66.2	
	252	40	0.235	72.3	0.225	69.2	0.195	60	
	253	60	0.241	74.2	0.235	72.3	0.215	66.2	
Fulfil	254	0	0.33	100	0.245	74.2	0.24	72.7	
	255	20	0.205	62.1	0.17	51.5	0.16	48.5	
	256	40	0.205	62.1	0.167	50.6	0.148	44.8	
	257	60	0.2	60.6	0.2	60.6	0.195	59.1	

Specimen Number	Expo- sure (Secs)	Times when measured post curing							
		0 Hours		24 Hours		48 Hours			
Distallite	258	0	0.32	100	0.275	85.9	0.26	81.3	
	259	20	0.235	73.4	0.25	78.1	0.235	73.4	
	260	40	0.235	73.4	0.245	76.6	0.225	70.3	
	261	60	0.245	76.6	0.26	81.3	0.238	74.4	
Visiomolar Standard	262	0	0.29	100	0.270	93.1	0.255	87.9	
	263	20	0.215	72.4	0.230	79.3	0.215	72.4	
	264	40	0.230	79.3	0.261	90.0	0.215	72.4	
	265	60	0.24	82.8	0.230	79.3	0.215	72.4	
Visiomolar Grey	266	0	0.345	100	0.345	100	0.316	91.6	
	267	20	0.249	72.2	0.245	71.0	0.265	76.8	
	268	40	0.24	69.6	0.255	73.9	0.26	75.4	
	269	60	0.235	68.1	0.265	76.8	0.26	75.4	
Visiomolar Brown	270	0	0.300	100	0.295	98.3	0.295	98.3	
	271	20	0.24	80.0	0.24	80.0	0.24	80.0	
	272	40	0.26	86.7	0.225	75.0	0.255	85.0	
	273	60	0.25	83.3	0.228	76.0	0.265	88.3	
Visiomolar Light	274	0	0.245	100	0.255	104	0.255	104	
	275	20	0.215	87.8	0.210	85.7	0.210	85.7	
	276	40	0.213	86.9	0.210	85.7	0.210	85.7	
	277	60	0.205	83.7	0.205	83.7	0.210	85.7	
Adaptic II	278	0	0.355	100	0.355	100	0.35	98.6	
	279	20	0.29	81.7	0.277	78.0	0.28	78.9	
	280	40	0.345	97.2	0.315	88.7	0.34	95.8	
	281	60	0.360	101	0.322	90.7	0.335	94.4	
Occlusin XL	282	0	0.32	100	0.3	93.7	0.305	95.3	
	283	20	0.215	67.2	0.21	65.6	0.21	65.6	
	284	40	0.220	68.8	0.215	67.2	0.21	65.6	
	285	60	0.215	67.2	0.205	64.1	0.205	64.1	

Specimen	Number	Expo- sure	Times when measured post curing					
			0	Hours	24	Hours	48	Hours
		(Secs)						
Occlusin S	286	0	0.410	100	0.405	98.8	0.4	97.6
	287	20	0.283	69.0	0.280	68.3	0.29	70.7
	288	40	0.279	68.0	0.271	66.1	0.300	73.2
	289	60	0.285	69.5	0.28	68.3	0.275	67.1
Occlusin DY	290	0	0.445	100	0.445	100	0.44	98.9
	291	20	0.360	80.9	0.360	80.9	0.365	82.0
	292	40	0.365	82.0	0.355	79.8	0.350	78.7
	293	60	0.355	79.8	0.360	80.9	0.360	80.9
Occlusin LG	294	0	0.38	100	0.39	103	0.39	103
	295	20	0.29	76.3	0.29	76.3	0.29	76.3
	296	40	0.288	75.8	0.29	76.3	0.295	77.6
	297	60	0.285	75.0	0.285	75.0	0.29	76.3
P-30 XL	298	0	0.270	100	0.265	98.1	0.270	100
	299	20	0.185	68.5	0.183	67.8	0.175	64.8
	300	40	0.185	68.5	0.180	66.7	0.180	66.7
	301	60	0.160	59.3	0.160	59.3	0.165	61.1
P-30 U	302	0	0.300	100	0.300	100	0.305	102
	303	20	0.245	81.7	0.245	81.7	0.24	80.0
	304	40	0.215	71.7	0.215	71.7	0.205	68.3
	305	60	0.230	76.7	0.230	76.7	0.221	73.7
P-30 Y	306	0	0.340	100	0.335	98.5	0.345	101
	307	20	0.220	64.7	0.215	63.2	0.195	57.4
	308	40	0.235	69.1	0.235	69.1	0.210	61.8
	309	60	0.250	73.5	0.243	71.5	0.235	69.1

Specimen Number	Expo- sure (Secs)	Times when measured post curing							
		0 Hours		24 Hours		48 Hours			
P-30 G	310	0	0.36	100	0.360	100	0.365	101	
	311	20	0.3	83.3	0.295	81.9	0.295	81.9	
	312	40	0.285	79.2	0.275	76.4	0.280	76.7	
	313	60	0.290	80.6	0.285	79.2	0.285	79.2	
Estilux Post	319	0	0.260	100	0.23	88.5	0.228	87.7	
	320	20	0.245	94.2	0.220	84.6	0.218	83.8	
	321	40	0.230	88.5	0.225	86.5	0.224	86.2	
	322	60	0.230	88.5	0.230	88.5	0.229	88.1	

## APPENDIX F. REFLECTANCE TESTS:

TABLES AND GRAPHS OF  
INDIVIDUAL RESULTS

5.3.4 Results for the reflectance tests conducted on individual resins.

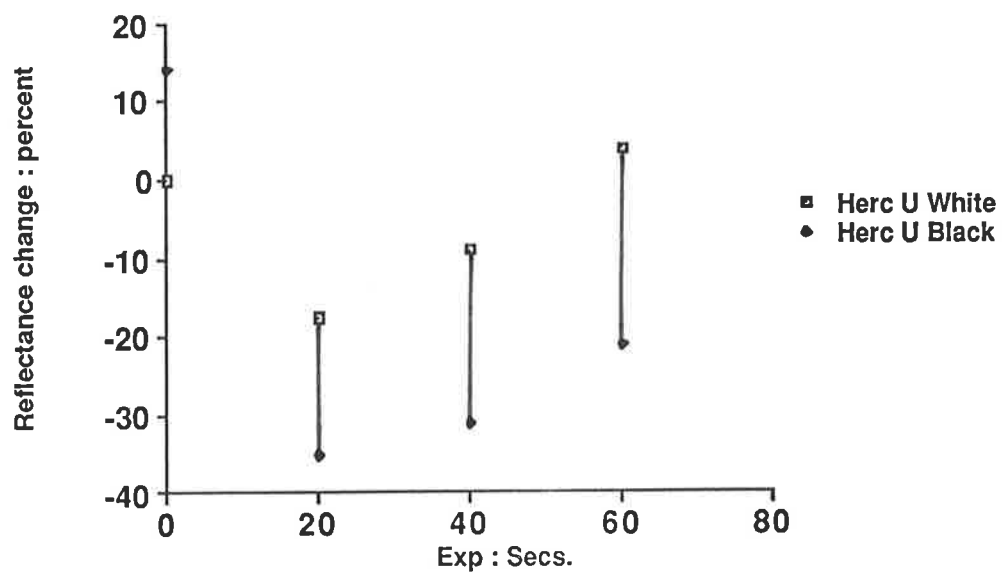
Resin Type	Specimen Number	Exposure Seconds	Candelas m <sup>-2</sup>		Percentage difference from white control	
			White	Black		
Herculite Universal	230	20	3.6	2.92	-17.5	-35
	231	20	3.46	3.02		
	232	20	4.05	2.74		
	233	40	3.6	3.09	-9	-31
	234	40	3.84	3.05		
	235	40	4.8	3.09		
	236	60	4.63	3.6	4	-21
	237	60	5.42	3.77		
	238	60	3.94	3.09		
	239	0	4.25	5.32	0	14
	240	0	4.80	4.80		
241	0	4.46	5.21			
Herculite Light	242	0	4.12	3.6	0	-12.5
	243	20	4.12	3.94	0	-16.5
	244	40	3.94	2.92	-4	-29
	245	60	3.81	3.05	-7.5	-25
Herculite Yellow	246	0	3.77	3.77	0	0
	247	20	3.46	3.25	-8	-13.5
	248	40	3.60	3.25	-4.5	-13.5
	249	60	3.94	2.78	4.5	-26.5

Resin Type	Specimen Number	Exposure Seconds	Candelas m <sup>-2</sup>		Percentage difference from white control	
			White	Black	White	Black
Herculite Grey	250	0	4.15	3.67	0	-11.5
	251	20	3.53	2.92	-15	-30
	252	40	3.6	2.74	-13	-39
	253	60	3.7	2.74	-10.5	-39
Fulfil	254	0	3.94	3.25	0	-17.5
	255	20	3.6	2.74	-8.5	-30
	256	40	3.63	2.85	-8	-27.5
	257	60	3.57	3.05	-9.5	-22.5
Distallite	258	0	3.94	3.29	0	-16.5
	259	20	3.46	3.26	-12	-17.5
	260	40	3.7	3.22	-6	-18
	261	60	3.6	3.19	-8.5	-19
Visiomolar Standard	262	0	5.15	5.66	0	10
	263	20	5.49	5.49	11	11
	264	40	5.83	5.49	11.5	11
	265	60	5.83	5.49	11.5	11
Visiomolar Grey	266	0	6.00	4.97	0	-17
	267	20	5.08	4.63	-15	-23
	268	40	5.08	4.56	-15	-24
	269	60	4.97	4.56	-17	-24
Visiomolar Brown	270	0	5.90	5.42	0	-8
	271	20	5.52	5.11	-6	-13
	272	40	5.21	4.87	-11.5	-17
	273	60	5.48	4.87	-7	-17
Visiomolar Light	274	0	6.11	4.70	0	-23
	275	20	5.73	4.52	-6	-26
	276	40	5.73	4.63	-6	-24
	277	60	5.56	4.52	-9	-26

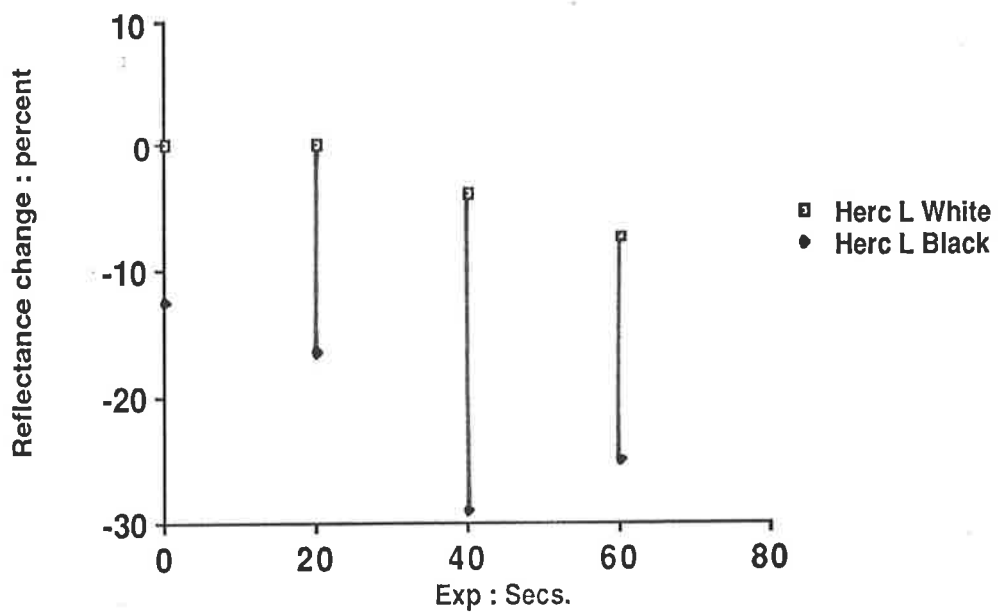
Resin Type	Specimen Number	Exposure Seconds	Candelas m <sup>-2</sup>		Percentage difference from white control	
			White	Black	White	Black
Adaptic II	278	0	5.08	4.63	0	-9
	279	20	4.97	4.18	-2	-17.5
	280	40	4.97	4.18	-2	-17.5
	281	60	4.97	4.18	-2	-17.5
Occlusin XL	282	0	3.77	3.67	0	-2.5
	283	20	3.77	3.70	0	-2
	284	40	4.46	3.94	18	4.5
	285	60	4.36	3.94	15.5	4.5
Occlusin S	286	0	3.94	3.57	0	-5.5
	287	20	4.12	3.70	4.5	-10
	288	40	4.05	3.77	2.5	-8.3
	289	60	4.12	3.77	4.5	-8.3
Occlusin DY	290	0	3.43	2.91	0	-15
	291	20	3.70	3.26	8	-5
	292	40	3.84	3.29	12	-4
	293	60	3.91	3.32	14	-3
Occlusin LG	294	0	3.94	3.32	0	-15.6
	295	20	4.29	3.77	8.5	-4.5
	296	40	4.46	3.94	13	0
	297	60	4.49	4.08	14	3.5
P - 30 XL	298	0	4.17	3.36	0	-19
	299	20	3.77	3.33	-9	-19
	300	40	3.77	3.26	-9	-21
	301	60	3.84	3.4	-7	-17.5
P - 30 U	302	0	4.08	3.32	0	-18.5
	303	20	3.94	3.43	3.5	-16
	304	40	4.12	3.5	1	-14
	305	60	4.12	3.43	1	-16

Resin Type	Specimen Number	Exposure Seconds	Candelas m <sup>-2</sup>		Percentage difference from white control	
			White	Black	White	Black.
P - 30 Y	306	0	4.49	3.70	0	-17.5
	307	20	4.53	4.15	1	-7.5
	308	40	4.63	4.18	3	-7
	309	60	4.46	4.15	-1	-7.5
P - 30 G	310	0	3.84	3.46	0	-10
	311	20	4.05	3.70	5.5	-3.5
	312	40	4.14	4.12	10.5	7
	313	60	4.32	4.18	12.5	9
Estilux Posterior	319	0	4.00	3.40	0	-15
	320	20	4.15	3.46	4	-13.5
	321	40	4.18	3.49	4.5	-12.5
	322	60	4.12	3.43	3	-14

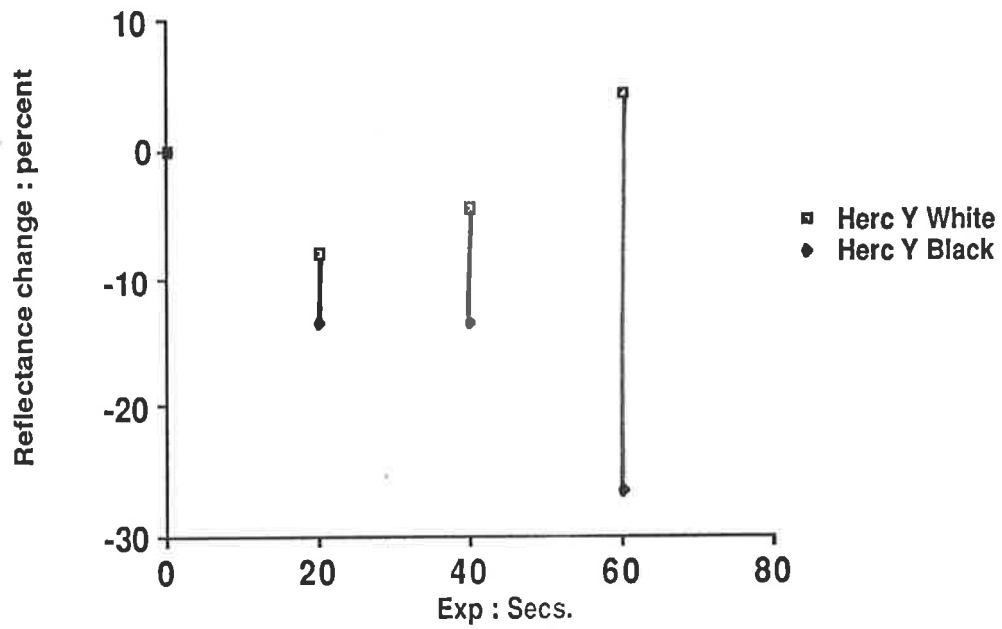
## Herculite Universal



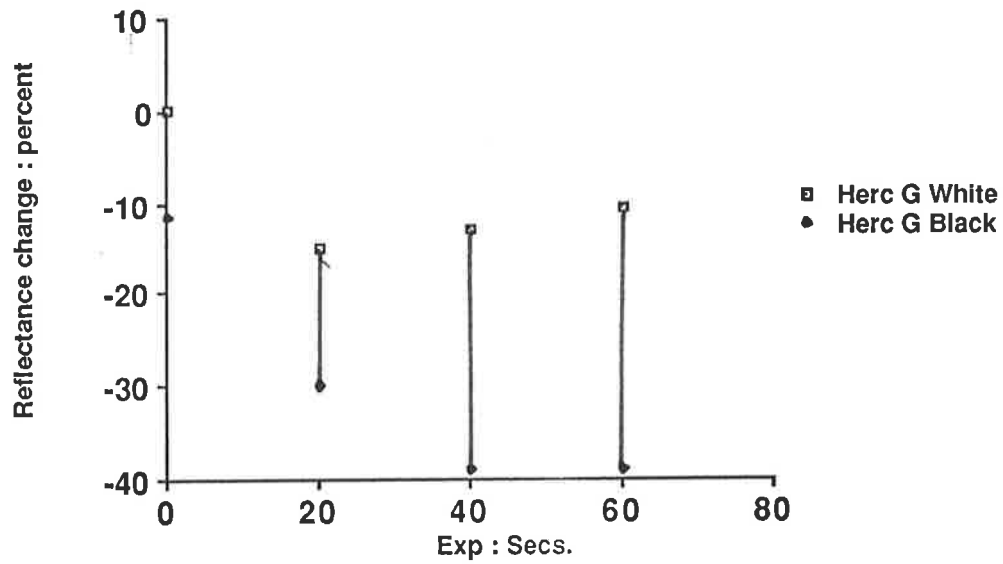
## Herculite Light



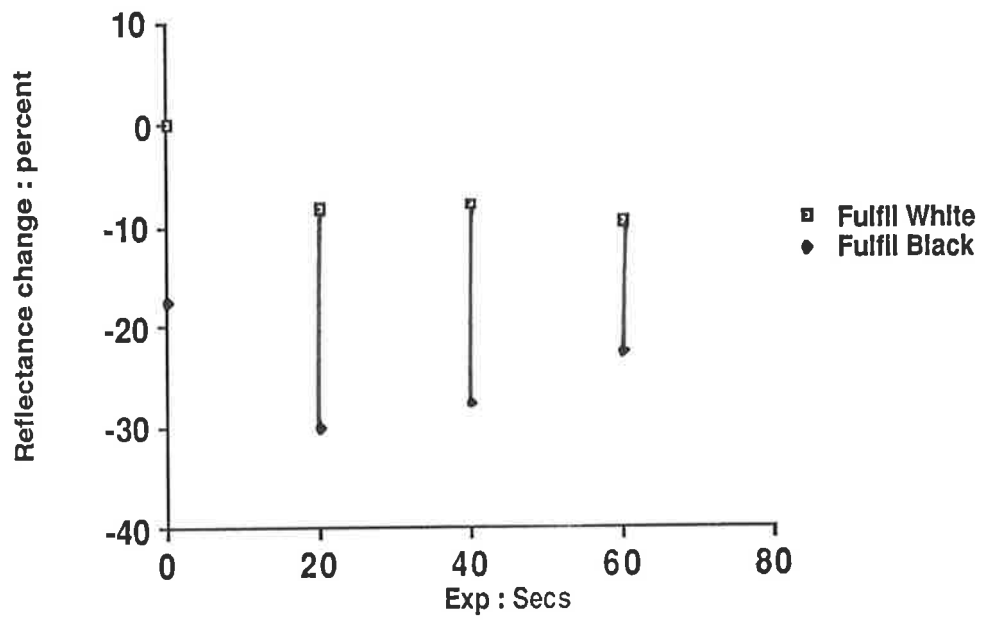
## Herculite Yellow



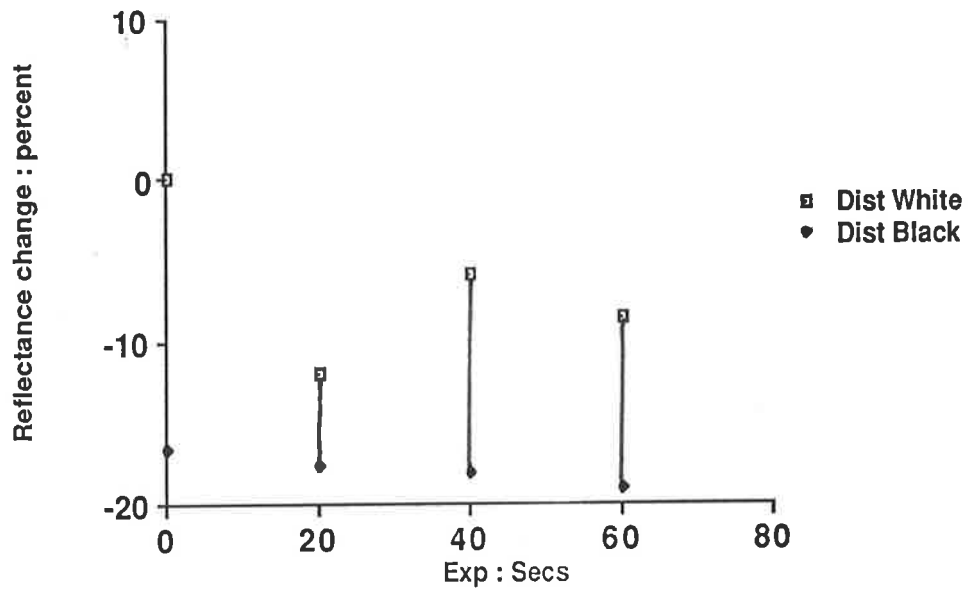
## Herculite Grey



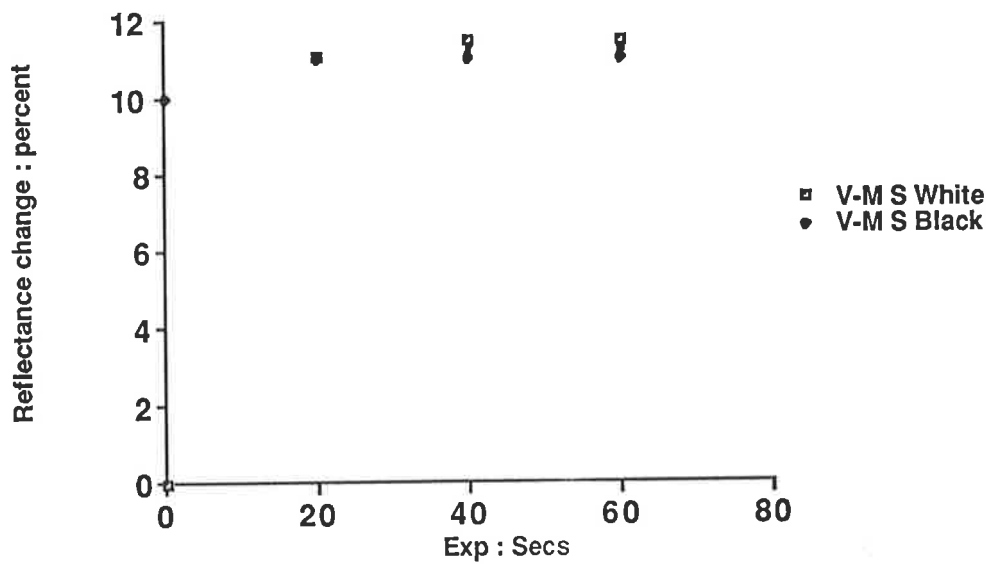
## Fulfil



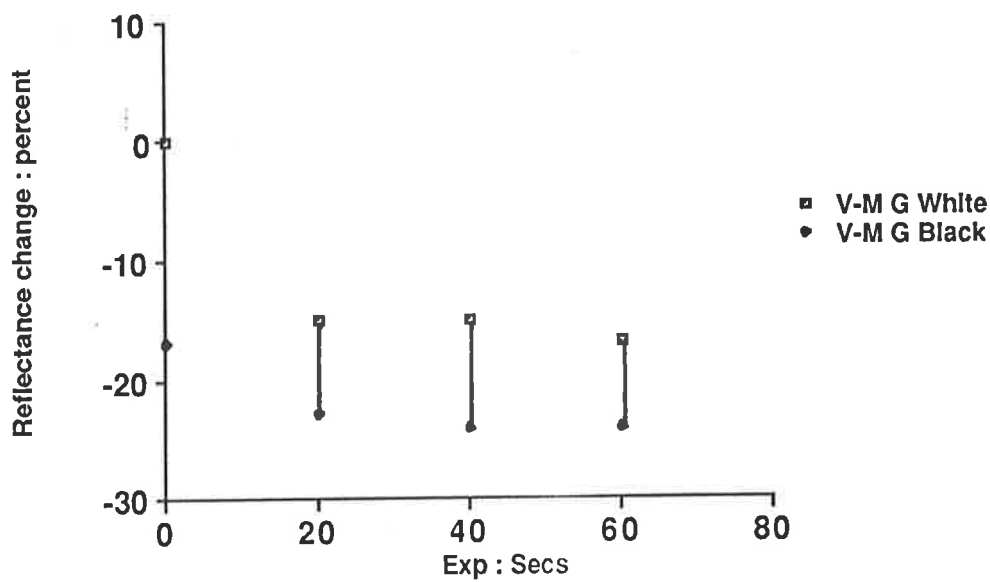
## Distallite



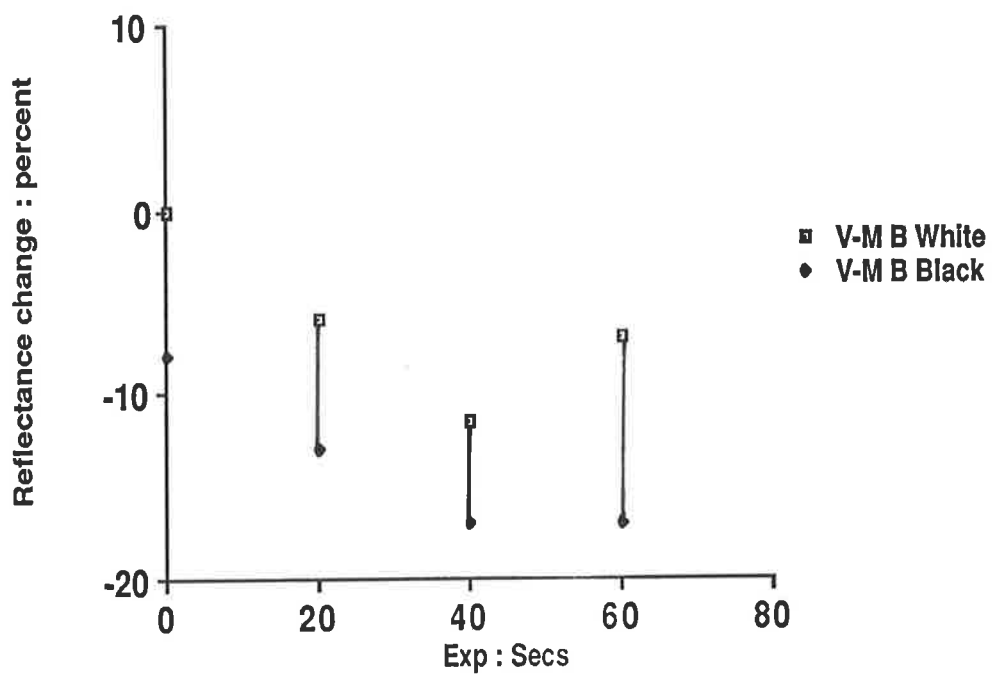
## Visiomolar Standard



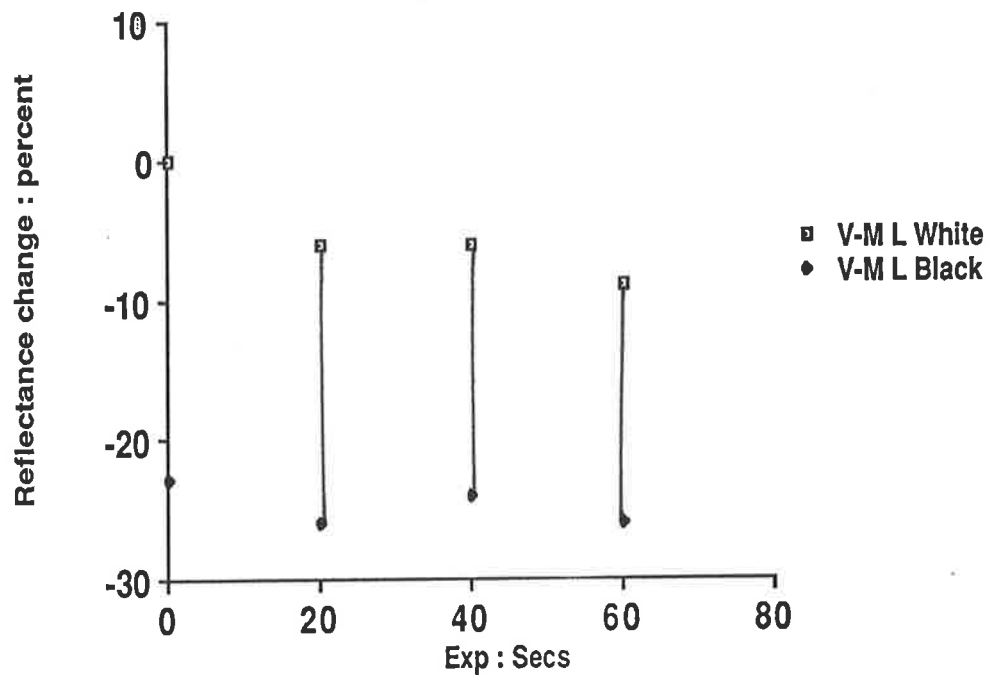
## Visiomolar Grey



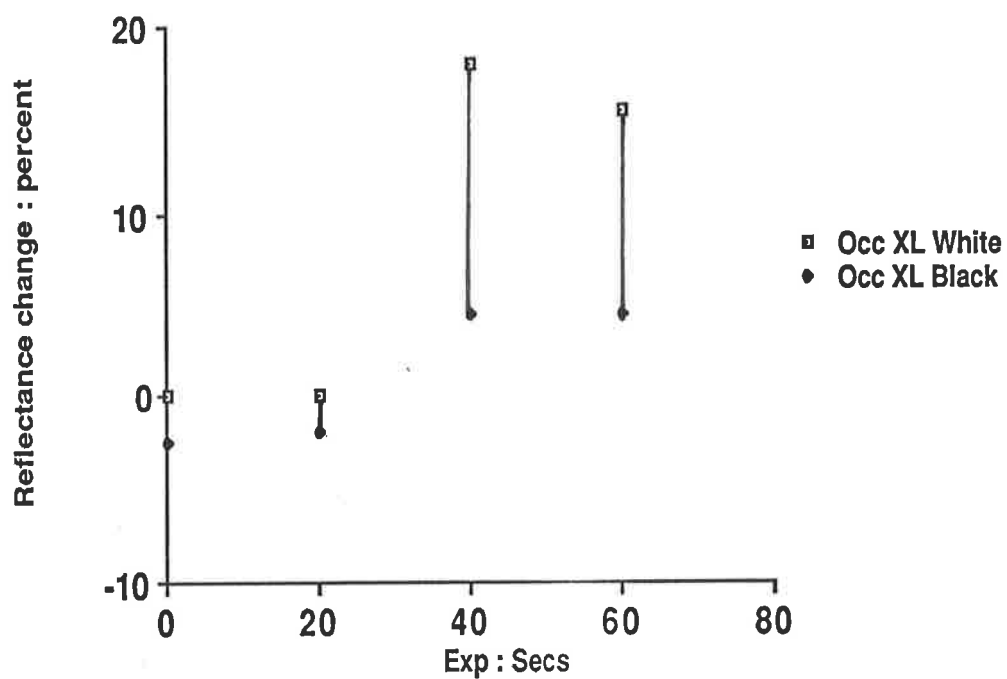
## Visiomolar Brown



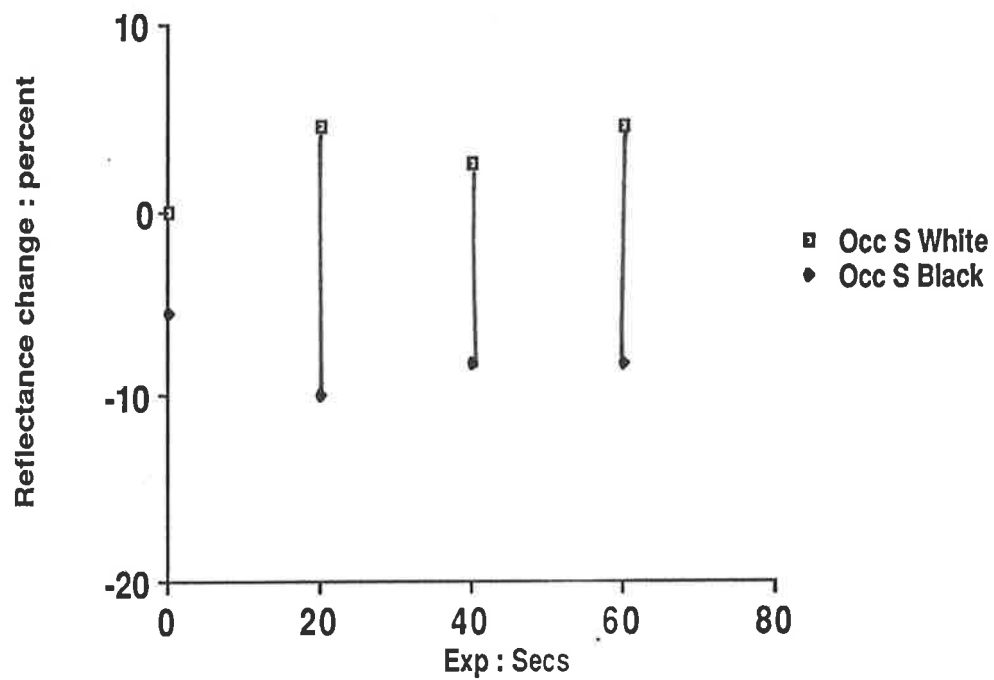
## Visiomolar Light



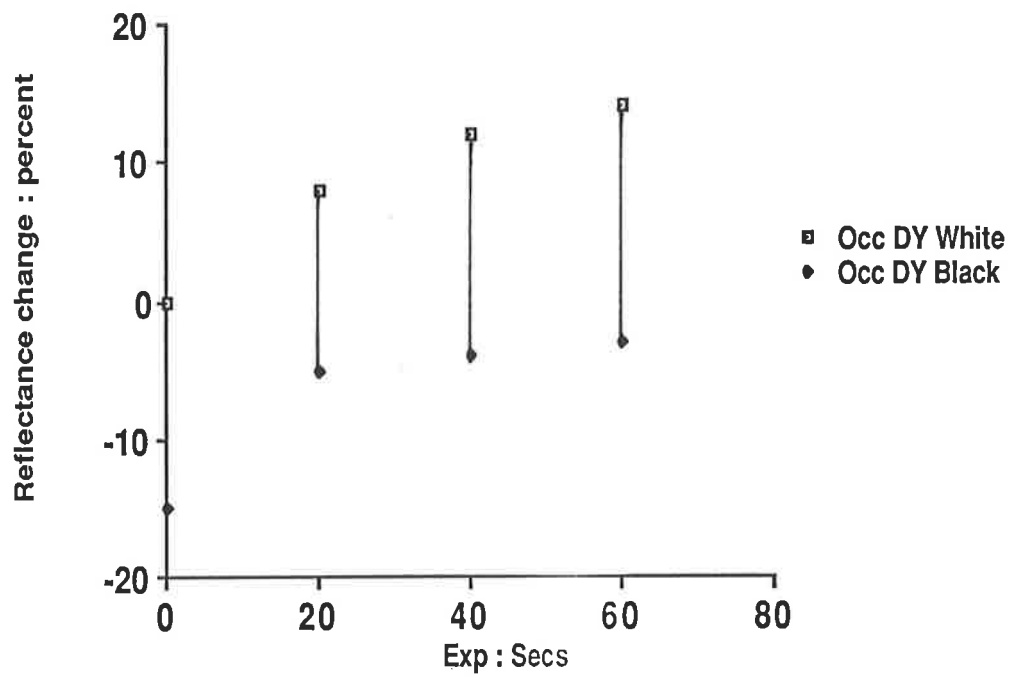
## Occlusin Extra light



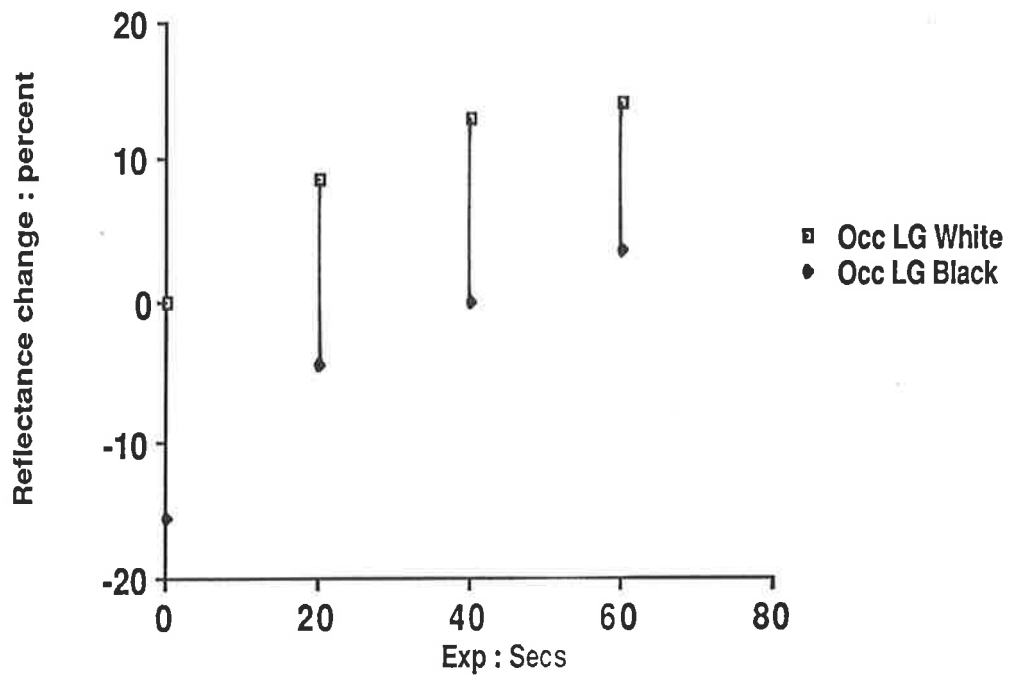
## Occlusin Standard



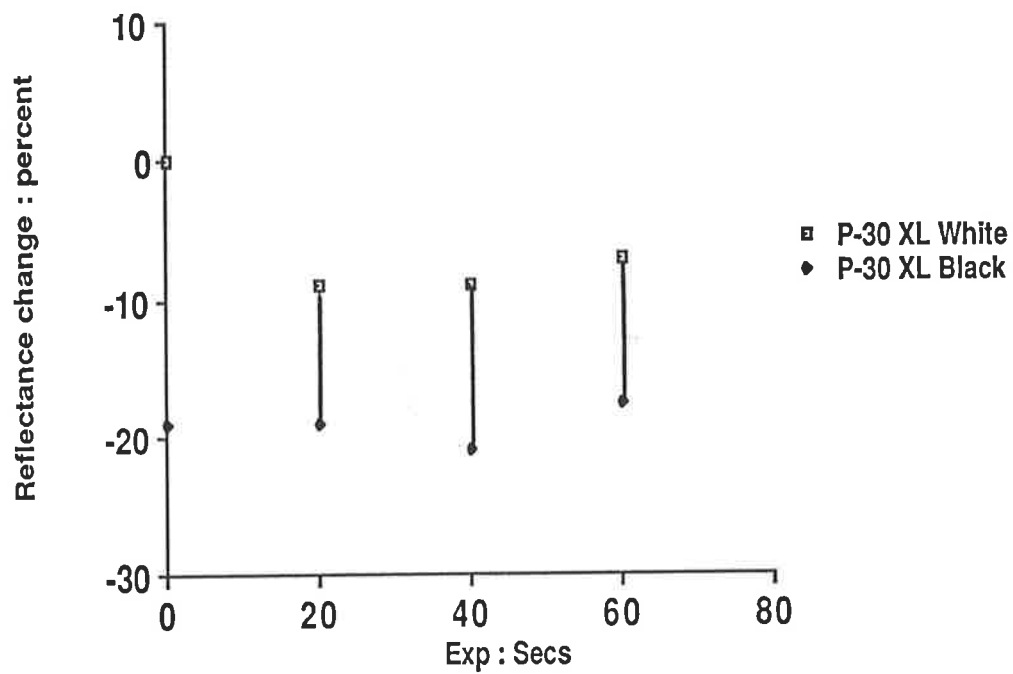
## Occlusin Dark Yellow



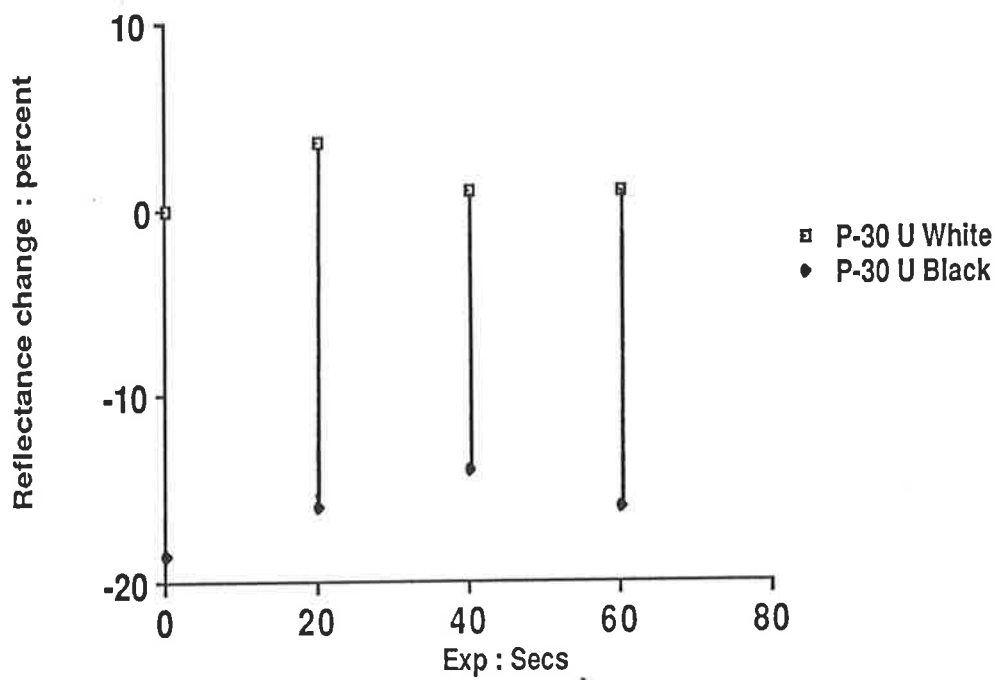
## Occlusin Light grey



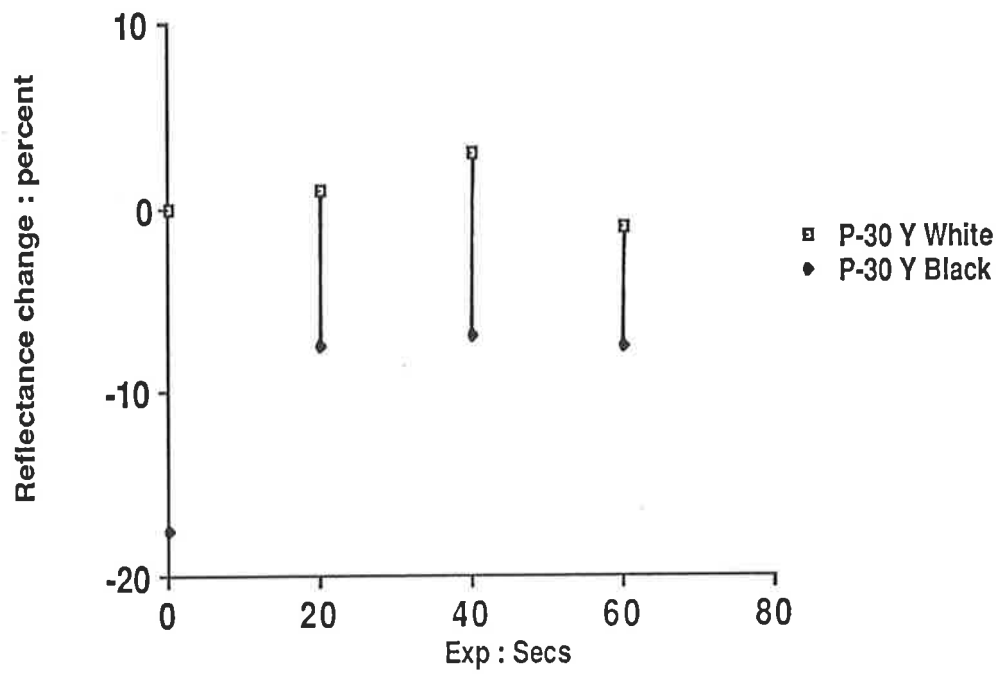
## P-30 XL (extra light)



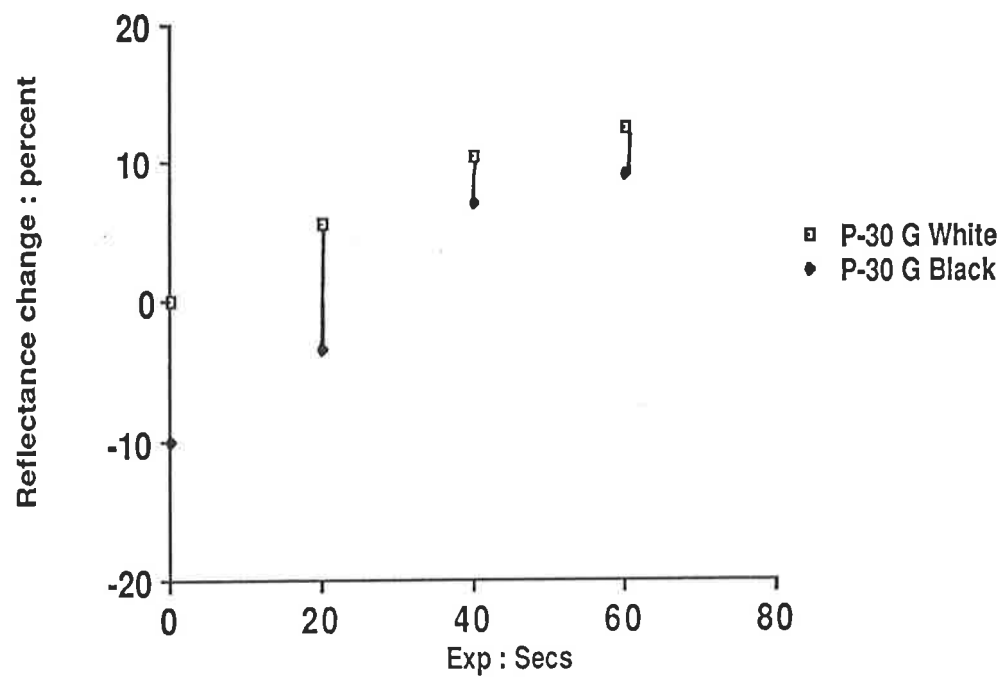
## P-30 Universal



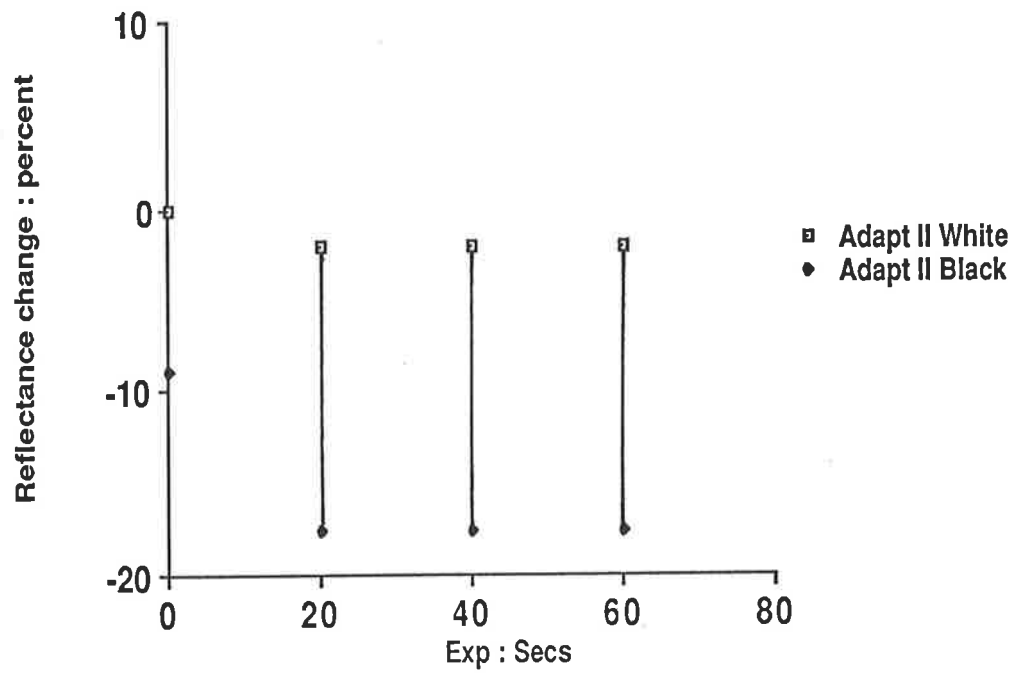
## P-30 Yellow



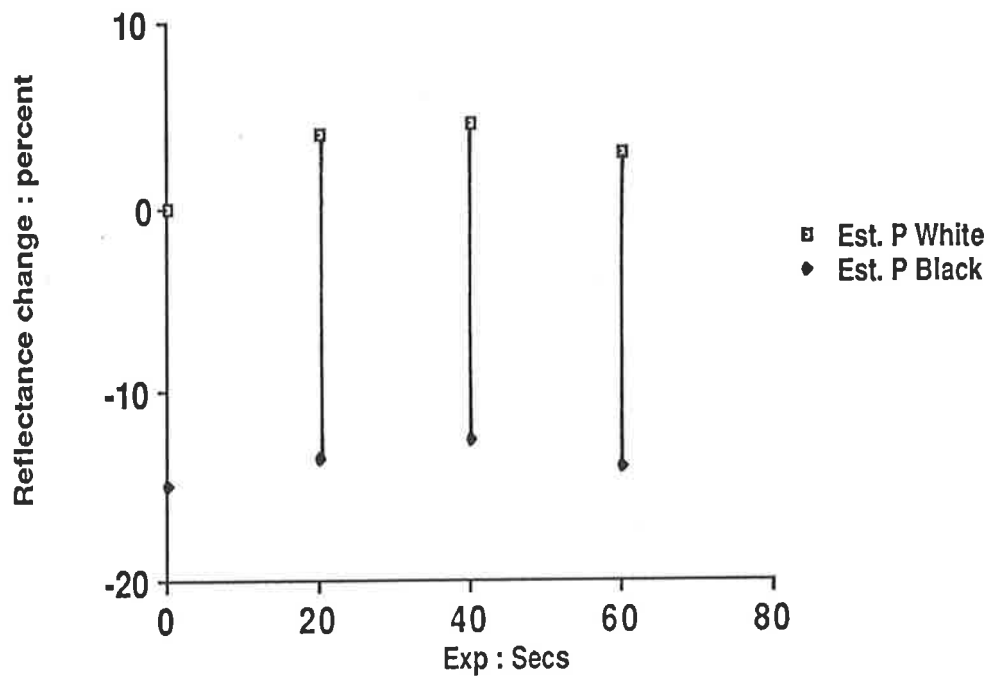
## P-30 Grey



## Adaptic II



## Estilux Posterior



APPENDIX G.  
CLINICAL PROTOCOL BY DR. R.J. SMALES  
USED FOR IN VIVO STUDY.

COMPARISON OF DISPERSALLOY WITH P-30, HERCULITE AND KETAC-SILVER  
AS CLASS I AND II CAVITY RESTORATIONS

R.J. SMALES - DEPARTMENT OF DENTISTRY

Purpose of Study

To assess the handling properties, clinical characteristics and longevity of a resin-bonded ceramic material (P-30; 3M C.), and a silver-reinforced glass-ionomer cement (Ketac-Silver; Espe), when placed in Class I and II cavities in the permanent posterior teeth of adults, at the Adelaide Dental Hospital.

Background

Traditional, metallic restorations used to restore posterior teeth have several disadvantages; poor appearance, tooth discoloration, marginal fracture, tooth weakening from tissue destruction needed for retention, extra appointments required for polishing and re-polishing later, lack of fluoride release, galvanism, the need for linings, and possible toxicity. Their chief advantage is in their resistance to wear and ease of handling. Until recently, resin materials have failed to withstand occlusal stresses over many years. However, newer generation materials have shown substantial promise, now that the probable mechanisms of resin failure are becoming understood.

The P-30 white-light cured resin is a highly-filled, densely-packed radiopaque material which has been used in clinical trials since late 1982. Wear rates have been reported as being similar to amalgam, and when used with

enamel etching and bonding, the restored teeth are then much stronger than when using amalgam.

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Herculite syringeable resin is also highly-filled and light cured, but the particle size is smaller than for P-30. Although Herculite has only been used in clinical trials since 1984, the initial results have been very good

Ketac-Silver has been shown to possess good in vitro abrasive resistance and good bonding to dentine and enamel and pins, and should possess adequate strength if confined to small class II cavities not involving cusp replacement. No clinical studies have been published, although the material has been in use for at least 2 years. Although not tooth coloured, the material has the advantage of producing minimal pulpal effects, of slowly releasing fluoride, and of bonding to dentine and enamel.

#### Preliminary Studies

All materials would be placed initially in posterior teeth in manikins, according to the manufacturers' recommendations, to gain experience in their handling. The material would then be immediately immersed in water for constant storage. A 'material handling form' would be completed by the operators, and the times noted for completion of similar sized restorations for each material (see attachment).

The filled tooth would be X-rayed, then the adequacy of the procedures assessed, using a rating scale method for different characteristics, e.g. surface texture, marginal adaptation, incomplete filling, porosities and retention. Then, after immersion in dyes, the tooth would be sectioned and assessed for marginal leakage.

## Subjects and Restorations

Informed written consent will be obtained from healthy adult patients, 445  
15-50 years of age, who volunteer to participate in the study. Attendance would  
be as part of the usual examination/treatment/recall system (see attachment).

Patients selected would have most of their dentition intact and need at  
least 4 restorations placed in vital teeth. The restorations would be in  
occlusion with the opposite teeth and should not involve cusps. Two-thirds of  
the restorations should be Class II, mainly in molars, and the remainder should  
be Class I, all in molars.

Some 50+ restorations of each of the 4 materials would be placed in  
approximately 30 patients, and followed up at 6 months, then yearly intervals  
for 3 years. Where possible no two test restorations will contact each other,  
and the materials should be placed in similar cavities chosen at random (using 4  
strips of cardboard). The materials should be inserted in the order chosen, from  
the distal to the mesial of the arches. There is no need to place more than one  
amalgam in each patient.

Safety and ethical considerations will be followed as given in the NH & MRC  
guidelines on human experimentation.

## Plan and Design

General data collection would include: name, age, teeth and surfaces  
treated, treatment used and date, operator, dates of recall visits, and  
reasons for any failures, with dates. A note will also be made of heavy  
smokers (more than 20 per day) and those with poor oral hygiene (extensive  
gingivitis).

2. Rubber dam will be used and the teeth wedged prior to cavity preparation. All pre-existing marginal staining will be removed and Life used as the routine lining under amalgam, and if there is less than 1.0 mm of remaining dentine when the other materials are used (see diagrams). Try and conserve all tooth cusps, and cut conservative preparations. 446
3. Traditional cavities will be prepared for Dispersalloy, and coated with, e.g. Copalite, prior to packing:
4. For P-30 and Herculite syringeable, the enamel margins will be bevelled (0.5 mm wide at 45°) after preparing minimal cavities. All dentine surfaces will be cleansed with 10% Citric acid for 5-10 seconds (or polyacrylic acid) before placing Ketac Fil as a base (see separate sheet for instructions). Condense the resins carefully, and white-light cure in 1.5 mm layer for 40 seconds. Also cure from the buccal and lingual surfaces after removing the matrices. (Keep the P-30 pots upside down when the lids are removed.)
5. For Ketac-Silver, the minimal cavities will generally not be lined, unless there is less than 1.0 mm of remaining dentine. The cavities will be conditioned using 10% of citric acid for 5-10 seconds (or polyacrylic acid), followed by thorough washing for 20-30 seconds, and drying. After placement, the restoration will be left isolated for 5 minutes from the start of mixing, before finishing under a water spray, using fine green and white stones.
6. After placement, all materials will be checked for occlusal interferences (using 2 layers of artic. paper) and gross excesses. At a subsequent appointment, finishing and polishing will be completed, using fine diamonds, greenstones, Sof-Lex discs, plug finishing burs, and prophy paste.

7. Each patient will have at least one Dispersalloy, but may have several P-30 and Ketac-Silver restorations.

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8. Any restorations lost during the study will be replaced with one of the alternative materials.

NOTE: The restorations must not be repolished once the study is underway.

### Analysis of Results

1. Baseline and recall records will be obtained (after marking contacts with arctic. paper) using: Ektachrome ASA 100 colour transparencies, taken at 1:1 magnification, using large Hu Friedy front-surfaced mirrors; and *polyvinylsiloxane* light-bodied impressions in Solo trays, from which diestone or similar casts will be poured. S.E.M. spluttered replicas may also be taken from the impressions.
2. The transparencies will be compared against Standard Sets of enlarged transparencies and scored on a linear scale, for such characteristics as surface staining, surface texture, margin staining, colour or translucency mismatch. Other features noted would include restoration fractured or lost, tooth fractured, tooth discoloured, general gingivitis or staining, and proximal contact adaptation.
3. The replicas will be compared against Standard Sets of replicas and scored on a linear scale also for marginal fractures, and surface loss of material (Lienfelder). Other features would include bruxism, restoration fractured or lost, tooth fractured, and anatomic form (including proximal contacts).

Some 30 restorations of each material will be replicated to test for examiner reliability.

4. Clinical data on failures would also be assessed, e.g. recurrent caries (with B.W.X.), repairs and fractures and patient problems, e.g. traumatic 448 occlusion, pulpal hyperaemia.
5. Data will be entered on The University of Adelaide VAX computer, and statistical analyses undertaken with the assistance of a statistician. It is hoped that meaningful results would be apparent within 2 years from the start of the study.

#### Support

Restorative materials have been donated and other material costs would be met from the research grant of Dr R. Smales. Chairside assistance for one half day a week is already provided for, but may need to be increased during University 'vacations' by mutual agreement.

Hopefully, this project would be an ideal one for a Dental Hospital Staff member, as a Clinical Diploma Candidate, to become involved with.

R.J. SMALES

18 February, 1986  
D105

## APPENDIX H.

## REPLICA METHOD USING ARALDITE D

1. Patient's impression should be cut to size and "boxed" with plaster (forming a tall well). The impression and plaster should be very clean.

2. Araldite mixture is made up using the disposable syringes:

(A)	Araldite M or D	100 ml
(B)	Hardener HY 964	100 ml
(C)	Accelerator DY 064	3.2 ml

Parts A and B are mixed together first in a denture cup by slowly rotating the cup (with lid) in your hands. This is done for at least 10 minutes and allows the minimum of bubbles to form. Once mixed this material can be left indefinitely in the refrigerator without fear of it polymerising.

Part C is added and mixed in the same fashion but will begin and accelerate the polymerisation. The material must be poured within the hour otherwise the viscosity will begin to cause problems.

3. Fill the mould only slightly above the impression material (enough to give a handle or grip to work with).

4. Vacuum infiltrate in the dessicator for at least 30 minutes. If too much araldite has frothed out in may need refilling.

5. Place in 60° oven (in Vicki's Lab) and leave for 48 hours. Check if material is tacky and if so continue heat treatment.

6. When the araldite has hardened, the plaster mould is carefully broken off and the araldite gently levered out of the impression material. Further trimming of the araldite block can now be done with either saw or file.

## APPENDIX J.

## 90° CONICAL DIAMOND.

This appendix contains the design and operating instructions of the Conical Diamond used in this project. This diamond has an inclusive angle of 90°.



## A Guide for Working with the MINILOAD-Hardness Tester

### Contents:

#### Technical Information

1. Description of the MINILOAD-Hardness Tester
2. Filling oil prior to performance of initial test
3. The hardness test (a) preparations for the test  
(b) producing indentations  
(c) evaluation of Vickers and Knoop indentations as well as of scratch widths
4. Exchanging diamonds

#### Directions for Working with the MINILOAD-Hardness Tester

5. Instructions for handling instrument
6. Preparation of test specimens
7. Instructions for operator
8. Physical deviations of correctly determined micro-hardness from macro-hardness
9. Instructions for scratch hardness testing

ERNST LEITZ G M B H WETZLAR

# Technical Information

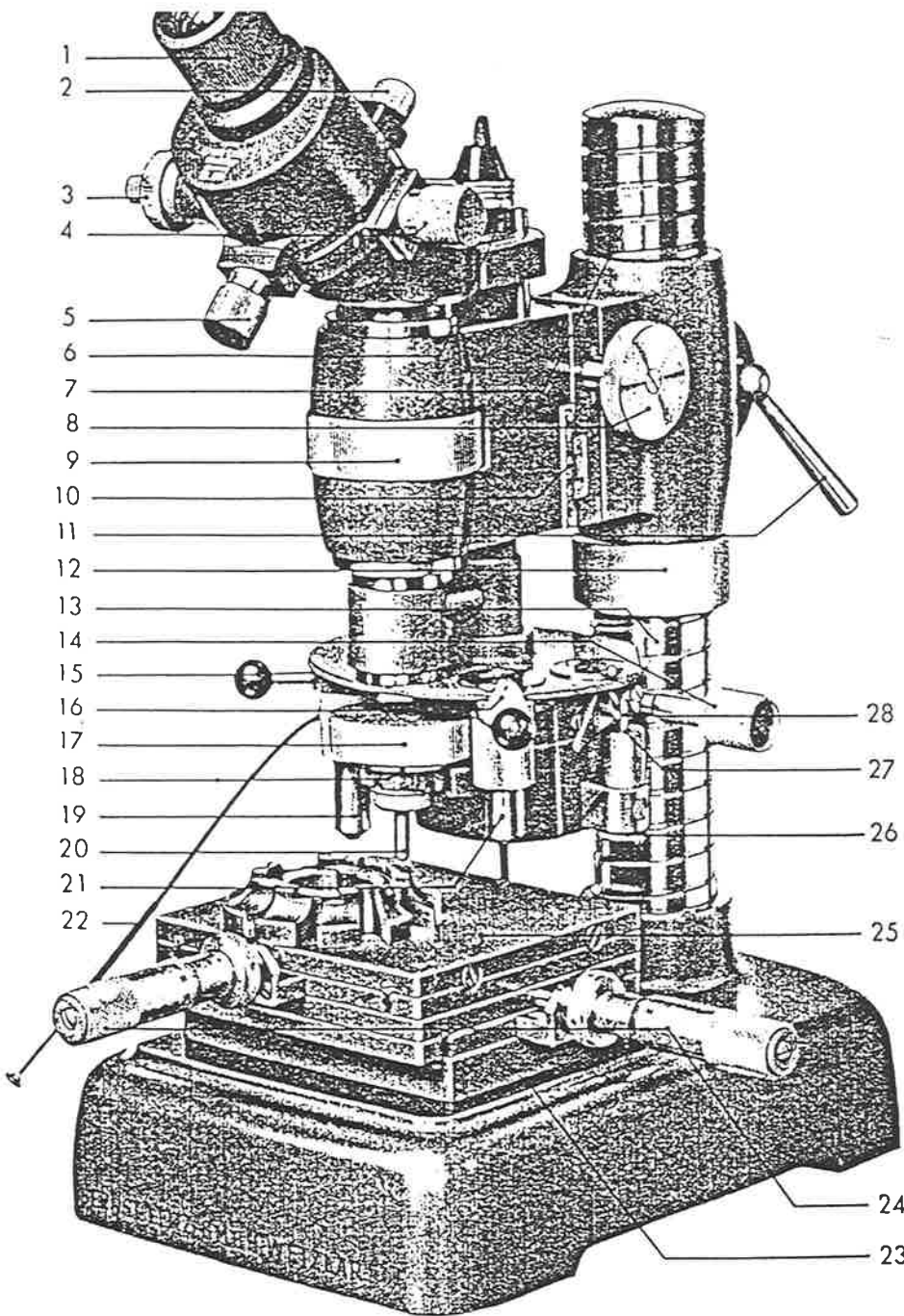


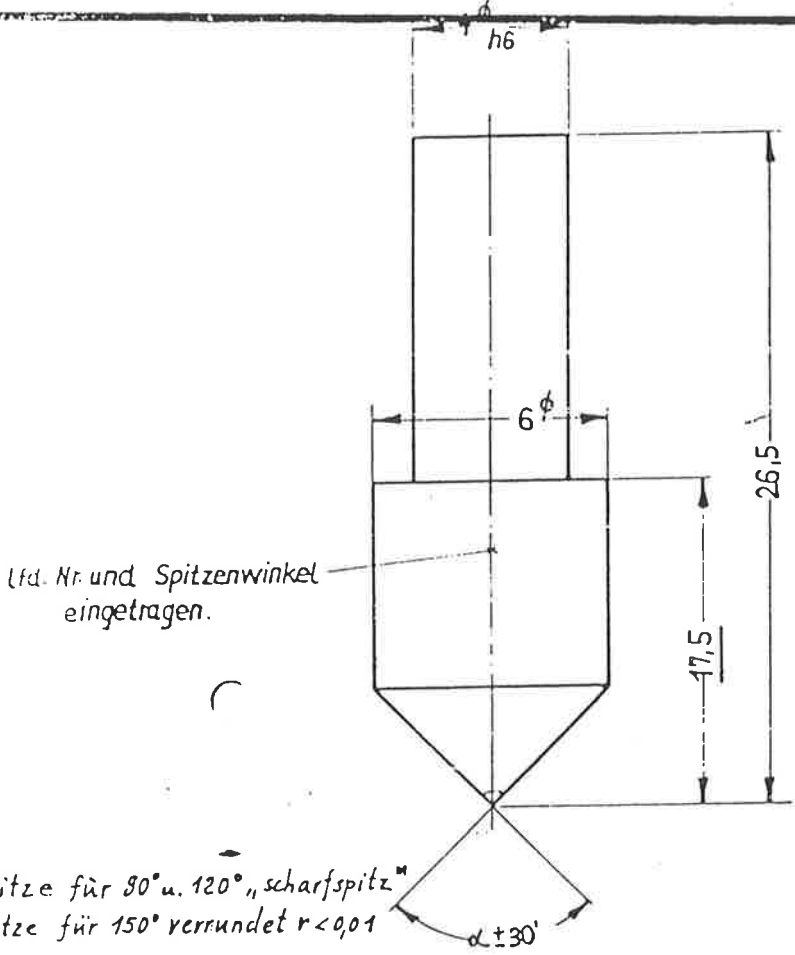
Figure 1

## 1. Description of the MINILOAD-Hardness Tester

1. Precision measuring ocular.
2. Ocular locking screw. After loosening this screw the ocular may be rotated about its axis.
3. Adjusting knob for graduated scale of precision measuring ocular.
4. Adjusting knob for transverse movement of ocular.
5. Adjusting knob for longitudinal movement of ocular.
6. Built-in removable illuminator with 8 volts, 0.6 amps incandescent bulb.
7. Index marking for normal position.
8. Knob for coarse vertical adjustment (range 40 mm).
9. Vertical micro-adjustment (range 14 mm).
10. Plotting scale with vernier for vertical adjustment.
11. Lever for clamping upper portion of MINILOAD-Hardness Tester to column.
12. Knurled ring for vertical adjustment of upper portion of MINILOAD-Hardness Tester on column (range 150 mm).
13. Upright column.
14. Knob with flexible shaft for raising indenter.
15. Handles for indexing objectives and testing diamond into position.
16. Ratched for locking objectives and testing diamond in indexed position.
17. Testing weight.
18. Loading disk for testing weight.
19. High power measuring objective (total magnification 400 x, diameter of field of view 0.45 mm).
20. Testing diamond.
21. Low power scanning objective (total magnification 100 x, diameter of field of view 1.8 mm).
22. Release for indenting mechanism.
23. Surface on which stage rests.
24. Micrometer spindles (range 25 mm).
25. Compound stage.
26. Knurled screw for adjusting rate at which diamond is lowered.
27. Lid with oil filling opening for hydraulic damper.
28. Piston rod.

... darf ohne unsere Einwilligung weder vervielfältigt  
 ch Dritten mitgeteilt oder überlassen werden. Sie darf durch  
 n Empfänger oder Dritte in keiner Weise missbräuchlich  
 wertet werden.  
 f die einschlägigen gesetzlichen Bestimmungen und Ihre zivil-  
 vlie strafrechtlichen Folgen wird verwiesen.

Ernst Leitz G. m. b. H. Wetzlar



Spitzenwinkel  $\alpha = 90^\circ, 120^\circ, 150^\circ$  bei Bestellung angeben!

Spitze zum Schäft laufend innerhalb 0,020  
 Gesamtgewicht 4,6g  $\pm$  20mg

~~Lieferfirma: Winter u. Sohn, Hamburg~~

Rohteil-Nr.	a	2x	siehe RM III/11/304		21.11.63	4.
Werkstoff magnetfrei, nichtrostend	Buch- stabe	Buchstabe kommt vor	Änderung		Tag	Name
LE Halbzeug	Maßstab	5:1	Tag	Name	Zeichnung Nr.	
	Gez.	28.2.62	Qi	60-379.024 (4)		
	Gepr.			früher 60-039.15 U 319		
Norm- geprüft	22.3.62					
Ernst Leitz G. m. b. H. Wetzlar	Benennung	Kegel-Ritz-Diamant				

... werden bei Abnahme besonders geprüft  
 ...

## 2. Filling Oil Prior to Performance of Initial Test

- 2.1 Before the MINILOAD-Hardness Tester is used for the first time, the hydraulic damping mechanism on the right side of the instrument is filled with oil. It must first be ascertained that the knurled screw 26 is turned clockwise till it stops. Using the pipette included with the instrument, the oil chamber is completely filled through the oil filling opening (marked in red) of the lid 27, till oil appears around the piston rod 28. Air bubbles should, as far as possible, be avoided when filling oil. Only the watch oil supplied by us with the instrument should be used.
- 2.2 Once the hydraulic damper is filled, the knob 14 for raising the indenter is turned clockwise till it stops, whereupon the release 22 is pressed, lowering the indenter. This operation is repeated till no further bubbles are visible. The knurled screw 26 is then adjusted to provide an indenting period of 15 to 20 seconds' duration. When temperature changes influence the viscosity of the oil, a re-adjustment is necessary, as an indenting period of less than 12 seconds' duration may produce inaccurate results.
- 2.3 The oil may be drained (for example for shipping or for cleaning the damping mechanism) by removing the screw 26, by turning it counter-clockwise, and raising the ball thus exposed with a pin. To drain it completely, the damper is operated several times.

## 3. The Hardness Test

### (a) Preparation for the Test

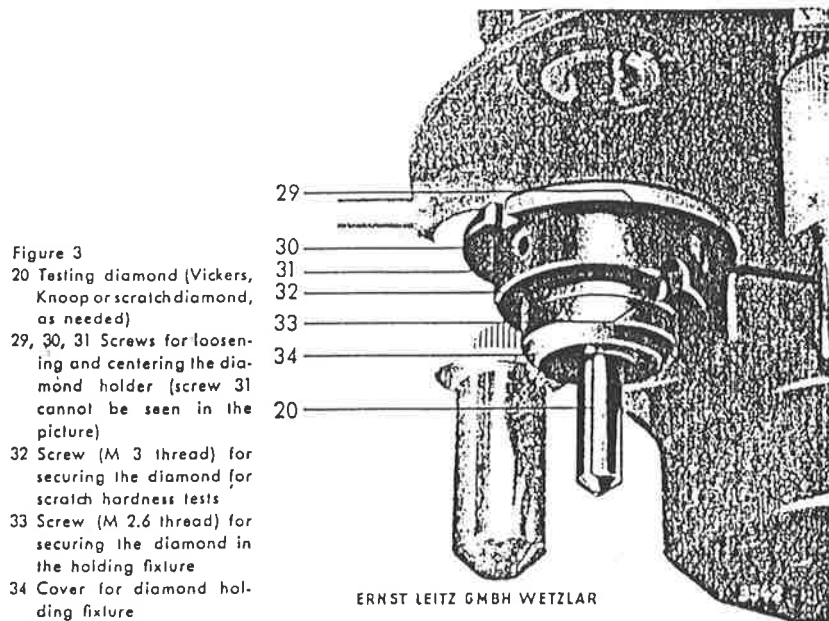
- 3.1 The transformer or resistance is connected to the house current, the illuminator switched on, applying 0.6 amps to the low voltage incandescent bulb.
- 3.2 The indenter is raised by turning knob 14 clockwise till it stops. This simultaneously cocks the driving spring.
- 3.3 The instrument is then adjusted, using the knurled ring 12, so that the specimen may be placed on the stage, or small parts may be held in the vice, directly below the indenter. The zero line of the vernier is then aligned with line 10 of the index 10 using knob 8, whereupon the MINILOAD-Hardness Tester is locked in place with the clamping handle 11.
- 3.4 Weight 17 is then placed on loading disk 18. Test weights for the following loads are supplied with the MINILOAD-Hardness Tester: 25, 50, 100, 200 and 300 grams. The indenting unit alone imposes a load of 15 grams  $\pm$  50 mg. Test weights of 500, 1000 and 2000 grams are available upon request. Consequently, tests may be carried out under loads from 15 to 2000 grams.

eg.  
 load of  
 400 grams  
 = indenter  
 of 15 grams  
 + weight of 385 grams.

- 3.6 By turning ocular 1 the reticule is brought into sharp focus,
- 3.7 whereupon the ocular scale is set at zero, with the aid of adjusting knob 3.
- 3.8 The image is adjusted, using coarse focus knob 8.
- 3.9 A suitable part of the specimen is centered in the field of view by manipulating the micrometer spindles 24.
- 3.10 Measuring objective 19 is then indexed (spring 16 locks it in place).
- 3.11 The image may then be brought into sharp focus with micro-adjustment ring 9.
- 3.12 The desired testing surface may be centered by turning the micrometer spindles 24.

(b) Producing indentations

- 3.13 The testing diamond is brought into indenting position by turning the revolver disk (spring 16 locks it in position).
- 3.14 Release 22 is then pressed gently and laid down, lowering the diamond slowly onto the specimen. Duration of this process about 15 seconds, see section 2.2.
- 3.15 Making Vickers and Knoop hardness tests:  
After the passage of an additional 10 seconds, during which the diamond rests on the specimen, knob 14 is turned clockwise till it stops, raising the indenter and simultaneously cocking the movement spring.
- 3.16 Making scratch hardness tests: (Please, see also instructions on pages 26/27).  
The micrometer spindle 24, located at the front of the instrument, is slowly and uniformly turned counter-clockwise (see section 9.5), moving the specimen toward the operator whereby the scratch is produced. Upon completion of the scratch knob 14 is turned clockwise till it stops.
- 3.17 The high power measuring objective 19 is then indexed (spring 16 locks it in position).
- 3.18 After loosening screw 2, the precision measuring ocular is rotated about its axis till the numbered graduations of the plotting scale are perpendicular to the diagonal to be measured, whereupon the knurled screw 2 is again tightened.
- 3.19 The cross hairs of the ocular (the intersection between the 75  $\mu$  line and the vertical line) are then centered upon the center of the indentation, using the adjusting knobs 4 and 5. Thus a reference point is obtained. Care should be taken that, when centering the reading scale is set at zero with the aid of knob 3.  
The following indentations may be produced with a positiveness of 2 to 3  $\mu$ .



#### Scratch Hardness:

(Please see also instructions on pages 23/24)

- 3.28 After loosening the locking screw, the ocular should be rotated about its axis till the numbered graduating lines of the plotting scale lie parallel to the edges of the scratch.
- 3.29 One edge of the scratch is brought in contact with a line of the plotting scale with the aid of knob 3 (see section 9.8), whereupon the measuring scale is read. The other edge of the scratch is thereupon brought in contact with the same or another graduating line and the measuring scale is again read, the difference between the two readings constituting the scratch width.
- 3.30 The test should be repeated using different loads so that by interpolation the specific load producing a basic scratch width of  $10 \mu$  is found. This load may then be considered as the scratch hardness.

#### 4. Exchanging diamonds

- 4.1 Only such testing diamonds as those which with their mountings are similar to the one supplied with the instrument in diameter, length and weight may be used. Weight: 4.6 grams  $\pm$  20 mg.

tations, so that when the diagonals are larger than  $25\ \mu$ , the MINILOAD-Hardness Tester is accurate within  $\pm 2-3\%$ .

## 9. Instructions for Scratch Hardness Testing - *see also Page*

- 9.1 The scratch diamond is inserted, observing the rules caution given in section 5.8. In order to block the slippage compensation, which is unnecessary for scratch hardness testing, the threaded pin 32, figure 3, accessible from the front of the instrument, is screwed in till resistance is clearly noticeable. As a result, the indentation made with the scratch diamond will no longer appear in the center of the field of view, but in the lower half of it, and may possibly lie outside it all together. For approximate centration of the indentation in the field of view, the forward micrometer spindle of the stage is turned approximately 0.5 mm counter-clockwise.
- 9.2 The hardness tester must, when used for scratch tests, be adjusted to the direction in which the stage is moved, by rotating it about the column. The marker on the microscope mounting must be exactly in line with the marker on the column.
- 9.3 If an angular scratch diamond is to be used instead of a conical one, it must be aligned with the direction in which the scratch is to be made. This is accomplished by first making a scratch with which the ocular graduation lines may be adjusted in parallel. Subsequently, one indentation only is made with the scratch diamond. When the indentation is then

brought into the field of view as directed in section 9.1, the position of the diamond relative to the direction in which the scratch is made can easily be seen; see figure 26. Upon loosening the threaded pin 33 shown in figure 3, the axis of the diamond may be rotated and aligned more precisely. The rules of caution given in section 5.8 should be observed.

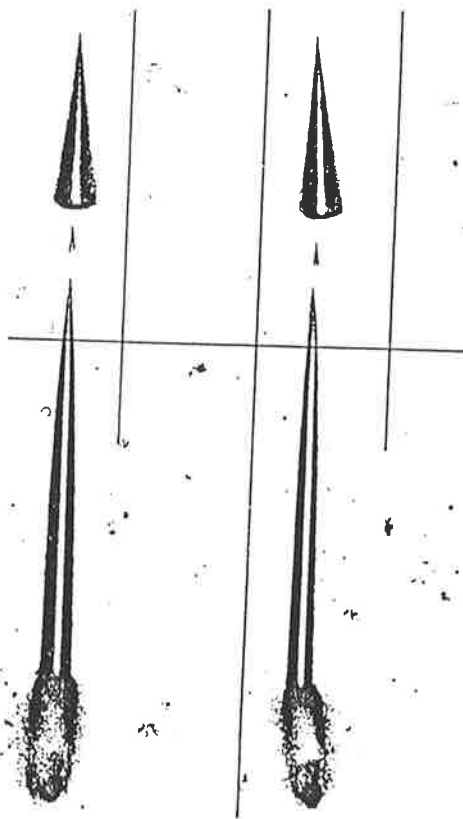


Figure 26  
Impression and scratch produced with a properly oriented scratch diamond (left) and one set at an angle (right). The impressions above are intended for orienting only and were therefore produced with a heavier load. The different scratch width resulting from incorrect orienting should be noted.

- 9.4 The specimen should be mounted and held in place as suggested in sections 6.1 and 6.2.
- 9.5 The scratch is made by turning the forward micrometer spindle of the stage slowly counter-clockwise. The duration of this motion should be approximately 10 seconds. A complete revolution in less than 5 seconds would produce incorrect results.
- 9.6 In order to avoid damaging the scratch diamond, specimens of unknown hardness should first be tested with the minimum load (15 grams). The scratch width should be approximately  $10\ \mu$ . It is, however, advantageous to produce wider scratches, up to  $20\ \mu$  in width, in order that the reading error of  $\pm 0.2\ \mu$  should constitute only 1% of the scratch width.
- 9.7 The scratch width is of value only for relative comparisons between various materials, the prerequisite being that identical types of diamonds are used and that all scratches are lighted by the same illuminating device when measured.
- 9.8 It is practical to align the edges of the scratch consecutively with the ocular graduation line. This may be clearly observed at the end of the ocular line.
- 9.9 The depth of a scratch made with a  $120^\circ$  scratch diamond is approximately  $\frac{1}{3}$  its width. The layer to be scratch-tested should be at least as thick as the scratch is wide in order to exclude the influence of the base on the test result.
- 9.10 From time to time it should be ascertained that the apex of the diamond is undamaged, as it is exposed to harm when scratching hard materials. For this purpose, an indentation is made in a soft metal, applying a great load. Any harm to the diamond can easily be detected by examining the indentation with the high power objective.
- 9.11 The diamond may become dirty as a result of its being touched or its coming in contact with a dirty specimen. Should this occur, it may be cleaned with a soft cloth and, if necessary, with lighter fluid.

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## APPENDIX K

## STATISTICAL COMPARISON OF DISC THICKNESSES

Number of discs	64
Thickness range	0.75mm - 1.141mm
Mean thickness	0.921 mm
Standard deviation	0.088

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