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SELECTED PROPERTIES OF HARD SETTING CALCIUM HYDROXIDE

by

Mohamed S. Hwas

A Thesis Submitted to the Faculty of the Graduate School of Loyola University of Chicago in Partial Fulfillment

of the Requirement for the Degree of

Master of Science

April

1983

DEDICATION

To my wife Mariam

My daughters

Nisreen and Rema

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VITA

Mohamed S. Hwas was born on December 20, 1950, in Zawia, in Libya.

He received his elementary and secondary education in the Zawia secondary school in Libya. He graduated from this school in 1970.

In 1970, he began his dental studies at the Faculty of Cairo School of Dentistry. He graduated in 1975, with the degree of Doctor of Dental Surgery.

He practiced general dentistry with the Government and as a private practitioner in Libya for a period of four years.

In 1981, he entered Loyola University School of Dentistry in a two year graduate program leading to a certificate of specialty in Operative and a master of sciences in Oral Biology.

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CHAPTER I

INTRODUCTION

The use of a base under restorative materials has been advocated clinically for many years. The base material provides protection for the dental pulp during restorative material condensation and acts as a thermal and electrical insulator. Of all the medicaments used for pulp protection, calcium hydroxide has, according to many investigators, been considered the best.

Calcium hydroxide is particularly effective in promoting the formation of secondary dentin. Secondary dentin is an important aid in the repair of the pulp. Likewise, it provides a thicker layer of dentin, which assists in protecting the pulp from irritants, such as toxic products from restorative materials or deleterious agents that may penetrate from microleakage. In very deep cavities the antibacterial properties of calcium hydroxide materials, coupled with their ability to promote dentin formation offer a distinct advantage.

Acid etching of tooth enamel is widely used to increase the retention of resinous sealants and restorative materials. The use of concentrated acid solutions on vital dentin may seriously aggravate the pulpal response to composite resins. A protective layer of calcium hydroxide containing base material to cover exposed dentin is frequently recommended.

An intermediate material should stimulate pulp repair. It should protect the pulp from injurious toxic agents found in restorative materials. The base should provide firm foundation for the support of the forces required to condense direct gold and large amalgam restoration. It should withstand solubility and disintegration in the oral fluid. It should have good physical properties and possess adequate antibacterial effect.

It was the primary purpose of this study to determine the water solubility of hard setting calcium hydroxide base materials as well as the solubility in 37% orthophosphoric acid.

Secondarily, a number of important parameters will also be studied, compressive strength, diffusion of calcium and hydroxide ions in an agar plate and antibacterial effect of hard setting calcium hydroxide base materials.

CHAPTER II

REVIEW OF LITERATURE

<u>Solubility</u>. Probably the one property of greatest clinical significance with regard to calcium hydroxide bases is solubility. The most common laboratory test for solubility is the measurement of disintegration in distilled water, as outlined in the A.D.A. specifications eight and nine.

Despite the long history and varied use of calcium hydroxide in dentistry the literature contains no reference to acid or water solubility <u>in vivo</u> or <u>in vitro</u>. This remarkable fact has been confirmed after exhaustive search of the literature. There was only one unpublished article about $Ca(OH)_2$ acid solubility by Dr. Swartz of Indiana University.

Within the past several years a relatively new form of this material (Ca(OH)₂) has been marketed; namely, so called hard setting calcium hydroxide. Our primary purposes are to investigate the properties of this new material.

The literature review of solubility will include those materials (other than $Ca(OH)_2$) are mainly used as bases under metallic or non-metallic restorations.

Wilson and Batchelor (1970) studied the solubility of zinc oxide-eugenol cement. Standard cement disks, 20mm diameter x 1.55mm thick were prepared by casting the paste into split-ring molds. The determination of zinc content of solutions was by atomic-absorbtion spectroscopy; eugenol was determined directly in aqueous solution by ultraviolet spectroscopy. They found that the disintegration of ZOE cements in aqueous medium is a consequence of the continual loss of eugenol, by leaching from the cement matrix. The cement thus loses its mechanical strength and disintegrates. They indicated that only long-term durability studies can give any indication of relative clinical durability of ZOE because of the variations in the changes of rates of elution of material experienced with different cements over a period.

Wilson, et al. (1971) examined the microstructure of zinc oxide-eugenol cement and the water-leached product by using electron microscopy and scanning electron microscopy. They concluded that zinc oxide-eugenol cement consists of a crystallite zinc eugenolate matrix that encases and binds the zinc oxide powder grains that have not been consumed in the reaction. Water hydrolyzes the zinc eugenolate to zinc hydroxide, which acts as an indifferent binder. Eugenol is completely leached from the cement. Thus the zinc oxide-eugenol cement is hydrolytically unstable and the degraded cement has little strength. Norman, et al. (1957) investigated the solubility of zinc phosphate and silicate cements in acids, pH of the media, and length of storage time. A circle of 10.4mm diameter was etched on a glass plate. A given quantity of cement was then dropped in this area. A stainless steel wire was immediately inserted in the tablet. Solutions of acetic acid, lactic acid, and citric acid of pH 4.0 and pH 5.0 were prepared. Ammonium hydroxide solutions at pH 8.0 and glass distilled water having a pH of 6.0 were also included in this study.

They found that the materials were more soluble in dilute organic acids than in distilled water. There was more dissolution of the materials in pH 4.0 than in pH 5.0 solutions.

Norman stated that the dissolution of cements tends to be minimized by tests utilizing prolonged storage in the same media. Zinc phosphate cements were markedly more soluble than silicate cements over a 1-week period when the specimens were placed in fresh solutions each 24 hours.

Norman, et al. (1959) continued the above research, the solubility of resins, cavity liners, zinc oxide and eugenol, and Kryptex (zinc-silicate cement) in various dilute organic acids and glass distilled water was investigated.

They found that silicate specimens continued to show marked dissolution in citric acid, pH 4.0, even after prolonged storage time. The resin materials tested were relatively insoluble; when the acid solutions were changed daily, an actual weight gain occurred presumably due to sorption of salts from the solution. Citric acid produced the greatest dissolution of the various materials. Mixtures of zinc oxide-eugenol were not quite as soluble in lactic acid or acetic acid as either zinc phosphate or silicate cements. Kryptex appears to be less soluble than zinc phosphate cement and more soluble than silicate cement.

The solubility of cements in the oral cavity has long been considered a primary cause of failure of cast restorations, contributing to recurrent caries and loss of retention. However, correlating clinical data to establish the relationship of the solubility in vitro with the relative rates of degradation in the oral cavity are limited. Mitchem (1967) was unable to detect, by clinical examination. differences in the behavior of silicate cement restorations prepared from mixes for which different solubility values had been obtained in the laboratory. Norman and associates, (1969) investigated the disintegration of zinc phosphate, silicate, and zinc oxide-eugenol cements. The cements were inserted in depressions located on the lingual surfaces of a cast partial denture framework, and loss of cement was monitored by weighing the restorations at specified time intervals. They concluded that the silicate cement showed the least disintegration, followed by zinc phosphate cement. A zinc oxideeugenol cement exhibited the most rapid loss of material. Analysis of the data indicated that under the conditions of the test the loss of material was a function of both the solubility and abrasion resistance of the material.

Richter and Ueno (1975) assessed the <u>in vivo</u> solubility of four cements inserted in 3mm diameter cavities placed in the pontics of three-unit fixed partial dentures. Four cavities were located in the

pontic so that they would be subjected to abrasion by the tongue and toothbrush, while four cavities were placed in such a manner that the specimens would not be abraded.

A data analysis indicated zinc silicophosphate cement had the lowest rate of disintegration, followed by zinc phosphate cement and zinc polycarboxylate cement, while EBA zinc oxide-eugenol cement had the highest rate of deterioration. There was significantly greater loss of material from regions where the cement was subject to abrasion than in those where abrasion was unlikely.

The only reported investigations on solubility of calcium hydroxide involve the so-called disappearing Dycal phenomenon. There was evidence of $Ca(OH)_2$ disappearing under restorations. Barnes and Kidd (1979) observed the loss of Dycal in four cases: they suggested a need for further studies. They proposed that the loss of Dycal beneath a properly sealed filling may not affect the prognosis of the tooth or restoration, particularly if the loss is caused by solution of the material and absorbtion into the pulp; of particular significance must be the clinical problem of distinguishing radiographically between a loss of lining and recurrence of caries.

The permeability of lining materials may be of decisive importance for their usefulness. Various substances have been used to assess permeability; including phosphoric acid, dyes, silver nitrate and radioactive isotopes.

Eriksen and Nordbo (1974) used the following materials to study permeability: calcium hydroxide, Dycal, a modified phosophate cement, a polystyrene liner and a copal resin varnish. The permeability was determined by penetration of water vapor through standardized discs of the lining materials.

They found that both Dycal and Copalite proved to be quite impermeable. Swartz and associates (1966) and Swartz et al. (1968) stated that both Dycal and Copalite were able to reduce markedly the penetration of P_{32} labeled phosphoric acid into the dentin from silicate and phosphate cement restorations. There was a slight increase in the permeability after treatment of Dycal with water or monomer which may indicate that the protective capacity of the lining materials can be reduced with time and after contact with free monomer from resinous restorative materials.

Wilson et al. (1981) showed that mixing calcium hydroxide with dimer acid made $Ca(OH)_2$ imprevious to water and allowed the material to set satisfactory under water. They described dimer acid as being a distillation product of hydrogenated dimerised oleic acid.

<u>Compressive Strength</u>. Studies of properties of cement bases used under restorations have dealt mainly with compressive strength and its implications clinically. Chong, Swartz, and Phillips (1967) showed that calcium hydroxide, ZOE, and zinc phosphate cement bases all have sufficient strength to withstand the forces of amalgam condensation without being displaced.

Farah, Hood and Craig (1975) used a computerized dental model to study the stress induced in a Class I amalgam restoration when supported by cement bases of various materials and thicknesses. They showed that the modulus of elasticity of cement base played an important role in determining the magnitude of both compressive and tensile stresses found in the amalgam.

Powers, et al., (1976) determined the modulus of elasticity and the compressive and tensile strength of commerical cements with primary and secondary consistencies by an optical strain gauge method. Powers indicated that of the cements of primary consistency tested, a non-eugenol zinc oxide cement had the lowest mechanical properties. A zinc polyacrylic cement base had the highest tensile strength, but a zinc phosphate cement base had the highest compressive strength and modulus of elasticity. A calcium hydroxide liner had higher mechanical properties than an unmodified ZOE liner.

They concluded that the strength required by base forming materials under amalgam restorations bears a relationship to the pressure exerted by the condensing instrument during the placement of amalgam. The pressure for amalgam condensation has been considered to be approximately 560kg/cm^2 (7952 psi). Bryant and Wing (1976) used eleven base forming materials under amalgam restorations to examine the rate of development of their compressive and tensile strength. Materials from three categories were compared, zinc phosphate cement, modified zinc oxide-eugenol cements, and calcium hydroxide cements. In the first study cylindrical test specimens (4mm diameter, 8mm high) were prepared in split stainless steel molds. Compressive and tensile strength were tested using Hornsfield Tensometer. In the second study cavities were prepared in extracted teeth and restored with amalgam after the placement of different base materials. Following sectioning, the restored teeth were examined microscopically.

They concluded that most base forming materials examined had reached a state of clinical set within five minutes from the beginning of mixing; however, Hydro-eugenol (a brand of ZOE) was set after two and a half minutes and Dycal after three minutes.

Up to at least ten minutes after mixing, zinc phosphate cements were considerably stronger than accelerated zinc oxide-eugenol materials which in turn were considerably stronger than calcium hydroxide materials whether the test was for compressive or tensile strength. On the basis of their clinical investigation, zinc phosphate cements, unmodified but set zinc oxide-eugenol cement, modified zinc oxide-eugenol cements and calcium hydroxide cements all possess sufficient early strength to be used successfully beneath amalgam restorations.

Plant and Wilson (1970) determined the early strengths of lining materials. The compressive strength of calcium hydroxide, carboxylate, zinc oxide-eugenol and zinc phosphate cements was determined. Specimens of lining material were prepared in silicone rubber molds and subjected to a compressive load on an Instron testing machine while remaining in the silicone molds. The temperature of the specimens is maintained constant in a mold at mouth temperature. The effect of moisture on a lining material while it is setting was investigated by placing saturated discs of filter paper above and below the specimen in the mold.

They showed that most of the materials were completely set in 7 minutes after the start of the mix. Materials older than 7 minutes are satisfactory, because their breaking stresses are greater than 10.5 N/mm² (1548 psi). However, Dycal and Hydrex showed much larger deformations of 10 percent and 7 percent respectively for 7 minute-old specimens when compared to the other lining materials.

They concluded that in the selection of a cavity liner, it must be remembered that success will depend upon the degree of set attained by the liner which is influenced by its age.

Vieira and Mondelli (1973) studied the fracture strength of Class II amalgam restorations condensed over protective bases. Tests were made in cobalt chromium dies of a lower right first molar containing a Class II cavity preparation. The base materials were: ZOE with 0.4 percent zinc acetate, Fynal Cement, Dycal, Zinc phosphate cement and copalite varnish.

They indicated that an amalgam restoration condensed over a zinc phosphate cement base showed no difference in fracture strength from those of the same restoration without a base or varnish. However, restoration over Dycal or ZOE under zinc phosphate showed a reduction in fracture strength.

Lloyld and Anderson (1980) investigated the strength and fracture toughness of calcium hydroxide preparation. To determine the compressive strength, cylindrical specimens (length: diameter = 2:1) were crushed axially. The materials used were Dycal, Pulpcap, Hydrex, Reocap, Hypo-cal and Calcipulp. The fracture toughness of the material was determined by fracturing single edge notch bars in three point bending.

All fragments were examined for porosity. Pores which appeared on the fracture surfaces of tension and compression specimens, which had radii greater than 10mm were measured and recorded.

They showed that the strength of Dycal is approximately twice that of Hydrex. The strength of Pulpcap and Hypo-cal is not quite as great as is implied by their toughness and pore size and is probably due to the closeness of largest pores to the surface.

They concluded that the strength of calcium hydroxide can be explained by considering both the toughness of the material and the pore size distribution. Dycal possesses the greatest actual strength in tension and compression. Suspensions of $Ca(OH)_2$ in water reportedly do not produce a set material with inferior strength to that produced by a chemical reaction.

Hormati and Fuller (1980) showed that the fracture strength of amalgam restorations depended on the base material and their thicknesses. Fracture strengths of Class II amalgam restorations decreased as the

thickness of the bases increased. Therefore, the strength of the restorations varied with the type of base. As a result of that zinc phosphate cement is equal to no base and superior to zinc phosphate cement plus Dycal, which is equal to Dycal only. Dycal is superior to Cavitec. The amalgam restorations were stronger over a base with a higher modulus of elasticity, modulus of elasticity is more important than compressive strength in supporting an amalgam restoration.

Lim and McCabe (1982) indicated that under moist conditions calcium hydroxide was able to withstand amalgam packing at 7 minutes. Moisture plays a major role in determining the early physical and mechanical properties of calcium hydroxide and its ability to withstand amalgam condensation.

Farah, et al. (1981) studied the effect of four cement bases; calcium hydroxide, reinforced zinc oxide-eugenol, polycarboxylate, and zinc phosphate on the fracture of amalgam.

The bases and amalgam were placed in Class I cavities prepared in the extracted teeth, the thickness of the bases varying from 0 to 3mm. The fracture load was determined by applying a compressive load at the rate of 0.2mm min⁻¹ to each restoration.

As the thickness of base was incrementally increased a gradual decrease is especially prominent for the bases of calcium hydroxide and reinforced zinc oxide and eugenol. Placement of a layer of calcium hydroxide covered by stronger base is often recommended in treating carious lesions close to the pulp.

The fracture load of amalgam supported by a zinc phosphate base, compared with calcium hydroxide, is not affected as seriously at 24 hours by an increase in the thickness of the base. The supporting ability of a base depends on its modulus of elasticity. At 24 hours the amount of load needed to fracture amalgam was lowered by 50% when the thickness of the calcium hydroxide base was increased from 0.5 to 1.0mm. Thus calcium hydroxide should be used only when necessary and limited to a thin lining.

Bacteriostatic Properties of Calcium Hydroxide

The practical significance of attempts to sterilize carious dentin by using medicaments beneath fillings lies in providing a case for the conservative treatment of deep carious lesions in which radical removal of all softened material is likely to cause mechanical exposure of the tooth pulp. Calcium hydroxide lining materials induce remineralization of the affected tissue and encourage defense mechanisms in the dentin.

McCue, et al. (1951) studied the antibacterial properties of silver amalgam, copper amalgam, gold foil, inlay gold, silicate cement, copper cement, zinc phosphate cement and quick-setting acrylic resin. Two pellets of each type of material were constructed, and duplicate platings were made throughout the experiments.

Three types of media employed in the experiments: nutrient agar, nutrient agar to which sterile serum was added, and nutrient agar to which sterile saliva from a non-carious subject was added. The test organisms used in the platings were twenty-four hour broth cultures of Micrococcus aureus, a gram-positive bacterium, and Escherichia coli, a gram-negative bacterium.

It was found that all the dental filling materials employed in the experiments produced inhibition to the growth of Micrococcus aureus and Escherichia coli. Silicate cement proved to be the most efficient material in inhibiting both gram-negative and grampositive growth in the three types of media. The other materials listed in their order of decreasing potency are copper amalgam, gold foil, zinc phosphate cement, copper cement, acrylic resin, silver amalgam, and gold inlay. The gram-positive organism was more sensitive to the bacteriostatic effect of the filling materials than the gram-negative bacterium.

Fisher (1972) investigated the viability of micro-organisms in retained carious dentin in permanent teeth. Ten vital permanent molars or premolar teeth with moderately extensive caries of the occlusal surface extending well into dentin were employed in this experiment.

It was found that viable micro-organisms are not recoverable from previously infected carious dentin in vital permanent teeth exposed to an initial lining paste of pure calcium hydroxide and water for a period of 6 months.

Fisher (1977) and Fisher and McCabe (1978) used Dycal, Kalzinol and Hydrex as lining materials. These materials were placed over infected carious dentin in vital permanent teeth for a period of 6 months.

Sterilization of the carious dentin occurred in all of these teeth exposed to Dycal, in seven out of nine where Kalzinol was used and in two out of nine of those employing Hydrex. It is considered that the antibacterial effect of Dycal results from its basicity and that Kalzinol acts by virtue of the prolonged presence of free eugenol. The difference between the effects of Dycal and Hydrex may be related to the opportunity for calcium hydroxide to gain egress from the set material.

Fisher and McCabe studied the difference in the antibacterial effect between Dycal and Hydrex by an examination of their chemical characteristics using infra-red spectroscopy.

The difference in the antibacterial capability of the two products must therefore be due to the difference in the ability of the calcium hydroxide to gain egress from the set material and produce an alkaline environment in the surrounding area, pH measurements using specimens of equal volume and surface area support this assumption. Two hours after placing a sample of Hydrex in a given volume of water the measured pH was 7.0 while that for Dycal was 11.5.

The antibacterial activity of Dycal and Hydrex on Oxford Staphylococcus, Lactobacillus casei and Streptococcus mutans was measured. Hydrex consistently showed no antibacterial effect against any of the organisms. Dycal and other calcium hydroxide brands like Reocap and Procal all showed clear antibacterial properties and ranked in that order.

McWalter, et al. (1973, 1976) exposed the pulps of 40 permanent teeth of monkeys and contaminated these teeth with saliva or plaque. The pulps were then capped with either Keflin (antibiotic), Durelon (a polycarboxylate cement), or Dycal. Varnish and then amalgams were inserted.

The findings of this study indicated that all of Dycalcapped pulp showed complete bridging and no inflammation - Durelon lacks an antibacterial effect and does not stimulate reparative dentin formation. Keflin proved to be too irritating to be of any value in pulp capping.

Dycal did not exert a persistent stimulating effect on the pulp that resulted in its eventual obliteration. Dycal did not induce a chemical pulpotomy and successfully supported the condensation of amalgam without the placement of any intermediary cement base. The compressive strength of Dycal is 1240 psi even after 24 hours in distilled water.

Olgrat, et al. (1973) compared the <u>in vitro</u> penetration of bacteria into the tubules of intact dental exposed by fracture in pairs of teeth, one of which in each pair was mounted with an intrapulpal hydrostatic pressure equivalent to 30mm Hg. The teeth were incubated at 37°C for 21 days. Intra-pair comparisons <u>in vivo</u> were made of bacterial invasion into dentinal tubules beneath ground dental surfaces and beneath fractures or acid-treated surfaces, which were exposed for 1 week. It was found that an outward fluid flow in the dentinal tubules due to intrapulpal pressure may mechanically hinder bacterial growth into the tubules. Of greater importance as an obstruction to bacterial invasion was the blocking of the outer opertures of the dentinal tubules by grinding debris. This barrier, however seemed to be removed after some days and this would allow bacterial ingrowth into intact vital dentin.

It is concluded that dentinal surfaces that have left unprotected and are covered with plaque for some days should be ground off to remove widened opertures filled with bacteria.

Mjor (1974) studied the presence of bacteria in experimentally exposed human coronal dentin in newly erupted teeth. He used 45 premolars, the teeth were demineralized in 5.2% nitric acid after grinding the enamel and dentin using diamond wheels and burs in high speed dental engine with ample water supply. The pulps were stained according to Brown and Brenn Technique for the demonstration of bacteria.

A bacterial plaque always covered surfaces which had been exposed to the oral environment for some time. Only slight penetration of bacteria into dentin was found, and the bacteria never reached the pulp.

Mjor (1977) showed that bacteria cannot be demonstrated subjacent to base and lining materials such as ZOE, $Ca(OH)_2$ mixed with water, or corticosteroids antibiotic materials. A few bacteria were found subjacent to two of seven unlined amalgam fillings and subjacent to three silicate restorations which had been lined with a polymeric material. Forsten and Karjalainen (1978) determined the bacterial growth in cavities after washing and by determining the effect of cavity cleaning agents on the microbial enzyme activity of carious dentin.

Bacteriological samples were taken after rinsing the cavity with water only, after experimentally infecting the cavity and after treating uninfected and infected cavities either with a saturated $Ca(OH)_2$ solution or with a chlorhexidine based detergent.

Cavities rinsed with water only showed sparse bacterial growth. The amino peptidase activity of carious dentin was inhibited totally with the $Ca(OH)_2$ solution, whereas the chlorhexidine based detergent had no effect on the enzyme activity.

Isermann, et al. (1979) in this study, a total of 32 teeth from two month old beagle male dogs were used to evaluate the influence of Dycal as a direct pulp-capping upon an exposed and bacterially infected teeth. The effects were evaluated with use of serial radiographs, consecutive vital dye injections, and histologic sections.

Eight of the nine bacterially infected exposed teeth were capped with Dycal showed significant amounts of vital pulp tissue with no noticeable periapical abnormalities. Whereas in all the teeth bacterially infected and exposed but not capped with Dycal, periapical lesions developed by the 15th day after exposure.

Leung, et al. (1980) and Fairbourn, et al. (1980) studied the effect of Dycal or Improved Dycal and IRM on bacteria in deep carious lesions.

They found that Dycal, Improved Dycal and IRM caused a significant decrease in bacteria in deep carious lesions when placed

in contact with the carious dentin for five months. Periera (1980) showed that no differences were detected in the pulpal responses to direct pulp capping achieved with a calcium hydroxide paste and with a calcium hydroxide powder. Law, et al. (1961) obtained the same result by using calcium hydroxide paste for the treatment of deep carious lesions in deciduous and young permanent teeth for a six month period.

Schwartzman, et al. (1980) evaluated the antimicrobial action of the following cements: zinc oxide-eugenol, polycarboxylate, zinc phosphate, silicophosphate and composite resin. The antimicrobial power of these cements was tested against, Streptococcus mutans, Escherichia Coli, Streptococcus viridans, Lactobacillus acidophilus and Streptococcus pyogenes. Blood agar plates were used to evaluate the antibacterial effect.

They observed that the cements had bacteriostatic and/or bacteriocidal action as listed in decreasing order of effectiveness; zinc oxide-eugenol, silicophosphate, zinc phosphate and silicate. Polycarboxylate and composite, exhibited no antibacterial action.

Vijinovic, et al. (1973) investigated the effect of citric acid treatment of cavities before insertion of a composite resin filling material.

Bacteria were apparent in numerous dentinal tubules beneath all the acid-treated cavities, but in only a few tubules under the untreated cavities. The pulpal reactions were stronger under the acid treated cavities.

Martin and Crabb (1977) reviewed the use of calcium hydroxide as an endodontic material. They indicated that calcium hydroxide provides a useful temporary root filling and may be used as a shorter antiseptic canal dressing as an alternative to one of the more familiar canal medicaments.

Mjor and Furseth (1968) studied which structural components in $Ca(OH)_2$ and corticosteroid covered dentin differed in mineralization from those seen in experimentally untreated or normal dentin by using an electron microscope.

In $Ca(OH)_2$ covered dentin the obturation of the tubules consisted of a moderately electron dense, crystalline material. In corticosteroid-covered dentin irregularly shaped tubules were observed close to the predentin and many tubules were obliterated by a highly electron dense material.

Sciaky and Pisanti (1960) determined whether the calcium from the calcium hydroxide dressing entered into the formation of dentinal bridging. Radioactive calcium was used to trace whether a diffusion of calcium ions occurs from the calcium hydroxide placed over amputated pulps into the dentin bridge of pulps of dog's teeth.

Forty two teeth were used, new dentin roofs were formed over the exposed pulps in every tooth examined. The presence of radiocalcium could not be demonstrated within the newly formed dentin roof over the amputated pulp. This study does not support the hypothesis that calcium hydroxide enters into the formation or calcification of a new dentin bridge.

Holland, et al. (1979) studied histologically the protection that hard tissue bridges offer to remaining subjacent pulp tissue after pulpotomy.

Complete hard tissue bridges produce a satisfactory protection to the pulp. In cases where the barrier is incomplete, inadequate protection was shown by the chronic inflammatory reaction in the remaining pulp tissue.

DeLaney and Seyler (1966) used hard set calcium hydroxide as a sole base in pulp protection.

Three hundred fifty-four cases were evaluated. When Hydrex was used as a deep liner, 93 percent of the cases were successful. When used as a direct or indirect pulp cap, 76 percent and 88 percent respectively were found successful.

Mechanism of Secondary Dentin Formation Due to Calcium Hydroxide

Calcium hydroxide was first used as a pulp capping agent by Hermann in 1930. Calcium hydroxide appears to provide a protective barrier for pulpal tissues not only by blocking patent dentinal tubules but also by neutralizing the attack of inorganic acids and their leached products from certain cements and filling materials. When placed over vital pulp exposures, calcium hydroxide stimulates the formation of reparative dentinal bridges.

Attalla and Noujaim (1969) using calcium hydroxide (Ca_{45}) as indirect pulp capping found that calcium 45 ions do not participate in the formation of reparative dentin. They indicated that the calcium in the reparative dentin does not originate from the pulp capping material, but from the systemic circulation. Mitchell and Walker (1958) studied the osteogenic potential of calcium hydroxide and other materials in soft tissue and bone wounds.

The osteogenic potential of calcium hydroxide has a rather unique potential to induce the formation of heterotopic bone when implanted in the connective tissues of the rat. The earliest appearance was 10 days and most of the specimens taken from 10 to 35 days gave this reaction. It is difficult to describe any importance to the pH of the material tested. Zander (1939), investigated the reaction of dental pulp to calcium hydroxide. He stated that the inorganic content of dentin, primarily a hydrated tricalcium phosphate, should follow the laws of mass action in relation to the solubility product. The blood is normally saturated, or super saturated with Ca and PO_4 ions, and hence any increase in the Ca or PO_4 ions, would cause precipitation or laying down of calcium salts. A material which contains either Ca or PO_4 in a combination which would be easily ionized when brought in contact with the surface of the pulp should react in this manner. This probably is the action of $Ca(OH)_2$. The normal mechanism of deposition of bone salts has been shown to depend to a large extent on the increase of PO_4 ions due to liberation of inorganic phosphate from blood or tissue by means of a phosphate enzyme. As bone phosphate is known to act best in an alkaline medium and as the solubility product decreases with increased alkalinity the condition here approach an optimum since $Ca(OH)_2$ has a pH of 12.4. Under these conditions there is a high pH and a large abundance of calcium ions and organic material as a matrix. For this reason a rapid precipitation of calcium phosphate might be expected.

In almost every case of vital pulp amputation a zone of structureless dentin is found on the outside of the dentin bridge, there is a dark, amorphous, structureless layer showing at some places cell enclosures on empty spaces. This probably is the result of the first chemical reaction between the calcium hydroxide and the tissue surface of clotted blood and degenerated injured pulp cells. Into this matrix, most probably, the first calcium phosphate precipitation takes place giving the proper base for the dentin formation. Dentin formation takes place not only in a healthy pulp but also in the presence of inflammation. Schroder, et al. (1971) stated that, calcium hydroxide is most widely used because it is believed to promote healing by induction of bordering hard tissue. Calcium hydroxide applied to amputed pulp causes superficial pulpal necrosis, underneath which hard substances formed. He added that Calcium hydroxide is very alkaline, pH about 12. When applied to the loose, connective tissue, it has a caustic effect with multi-layered necrosis. As a result, the multi-layered necrosis may be regarded as the combined effect of caustic injury, application pressure and increased intra pulpal pressure by edema, i.e., a mechanico-chemical effect.

The concentration of the hydroxyl ions rather than the calcium hydroxide as such is decisive for the initial tissue changes. Other hydroxides with roughly the same degree of disassociation would probably give similar results provided that the cation in question is not per se injuries. It has been suggested that magnesium hydroxide would have an effect similar to that of calcium hydroxide.

It is now known what initiates production of matrix. But, it is hardly any unique property of the calcium hydroxide for the induction of ectoptic bone formation. It was assumed that calcium hydroxide accelerates healing by suppressing the irritative effect of necrosis on the underlying pulp. The firm necrotic tissue acts as a mild irritant, to which the underlying tissue responds with bordering tissue formation. Once the pulp has been shut off from irritation, the tissue formation ceases, after which the newly formed tissue assumes the appearance of dentin. Matrix production was seen after four days, mineralization of tissue formation in the form of a bridge was observed after seven days. The first formed hard substance had an irregular bone-like apeparance, while the last one resembled dentin.

Bramnstrom, et al. (1976) studied the effect of $Ca(OH)_2$ and fluorides on human dentin. Intra-pair comparison of the dentin under cavities with $Ca(OH)_2$ dressings and control cavities showed differences in 5 of 11 pairs of teeth. Certain dentinal tubules directly under five $Ca(OH)_2$ treated cavities showed a varying degree of constriction. This change consisted in places of netlike structures or an amorphous substances. In certain tubules an increased number of calciospherites occurred on the walls of the tubules.

This investigation would suggest that only in certain cases can $Ca(OH)_2$ contribute to some constriction of the dentinal tubules. The SEM investigation also showed that great variations could exist from one dentinal tubule to another and that this change was confined to an area immediately beneath the cavity.

A $Ca(OH)_2$ dressing applied for a period of two months cannot be considered to give reliable obliteration of the dentinal tubules. This should be kept in mind when taking into account the fact that such treatment is considered to contribute to an obliteration of the dentinal tubules and thus give pulpal protection.

Shubich, et al. (1978) investigated the release of calcium ions from pulp capping materials. They showed that clinical and experimental evidence indicated that calcium hydroxide preparations placed over an exposed pulp cause healing by the production of reparative dentin.

Theories attribute this action of calcium hydroxide to either that of creating a favorable pH environment for calcification, or for providing various concentrations of calcium and making it readily available for diffusion into the pulp for incorporation into the forming reparative dentin.

The differences in the patterns of release of calcium could be explained on the basis of the composition of the products. Calcium

hydroxide contains other compounds that are added as fillers, binders, and catalysts to aid the set and ability of the product when it is manipulated for dental use. These added ingredients may affect the calcium ions that are released into the solution.

Because of these possible release factors, perhaps the difference of opinion in the possible sources of calcium in the formation of reparative dentin may be a result of such release considerations.

Torneck and Wagner (1980) reported the effect of Ca(OH)₂ on early cell division in the pulp. They showed that, a greater number of cell divisions was recorded in the calcium hydroxide restored teeth than in those teeth restored without calcium hydroxide. This indicated that calcium hydroxide as a cavity base has a stimulating effect on the repairing pulp and promoted cell mitosis. This stimulating or mitogenic effect may be associated with the local tissue pH and the presence of calcium ions, which disassociate from the aqueous calcium hydroxide paste and diffuse throughout the dentinal tubules, coming in contact with the pulp. The mitogenic effect produced by low concentrations of ionic calcium has already been recognized in other systems and tissues and has been related to its participation of DNA synthesis. If a similar action occurs in the dental pulp, then the therapeutic effect of calcium hydroxide liners may be derived, at least in part, from their ability to provide a greater number of cells for participation in the repair process. Whether these and existing cells are in turn stimulted to metabolize more rapidly has yet to be determined.

Eda (1961) studied histochemically the mechanism of dentin formation in the pulp, due to calcium hydroxide. Owing to the strong alkalinity of calcium hydroxide, the layer in the pulp contacting the paste becomes necrotic. In this necrotic layer granules consisting of calcium carbonate appear as a result of the combination of the calcium from the paste and carbonic acid. The pulp is protected by these layers of necrotic tissue and calcium carbonate granules. Meanwhile, in the vital pulp near the layer of necrosis, appear extremely fine particles originating from tissue fluid. They deposit and form a layer, in which as time elapses, magnesium and phosphate deposits, followed by the increase of odontoblasts resulted by metaplasia of the pulp cell lead to the completion of the dentin formation.

Finally, calcium hydroxide appears to be the most successful agent in inducing secondary dentin formation. Calcium ions deposited in the secondary dentin seems to come from the circulatory system. It is found also that calcium hydroxide accelerates the rate of secondary dentin formation. Whether the calcium hydroxide pH or its hydroxyl group has the principle mechanism in the dentin, formation is not clear yet. But, there is usually necrosis under the Ca(OH)₂ and then new dentin formation.

The primary aims of this study were to investigate, <u>in vitro</u>, the water and acid solubility of hard setting calcium hydroxide; also, to determine the compressive strength, calcium and hydroxyl ion diffusion and bacteriostatic effect of these materials.

CHAPTER III

MATERIALS AND METHODS

The materials included in the study are listed in Table I. Hydrex and MPC was used in calcium and hydroxyl ion diffusion only. All of these materials consist of two pastes which harden when mixed together. The pastes are contained in collapsible tubes, one of which is usually designated as a base paste and the other as a catalyst. The materials were proportioned in accordance with the respective manufacturer's directions either by weight or by dispensing equal lengths of pastes.

Table I

Material	Batch No.	Base/Catalyst Ratio	Manufacturer
		4 99 - 1999, <u>1999</u> - 1997 -	
Dycal	081782	1.2:1	L.D. Caulk
			Milford, Delaware
Life	22060	1:1	Sybron/Kerr
	21201		Romulus, Michigan
Procal	21871	1.15:1	
			St. Paul, Minnesota
Renew	052582	1:1	S.S. White
			Philadelphia
			Pennsylvania
Hydrex	07010		Sybron/Kerr
•			Romulus, Michigan
MPC	74062		Sybron/Kerr
	71132		Romulus, Michigan

Calcium Hydroxide Base Materials

<u>Solubility</u>. A modification of the American Dental Association Specification No. 8 for zinc phosphate cement will be used to study the water solubility of hard setting calcium hydroxide.

An adequate quantity of $Ca(OH)_2$ was placed in a stainless steel ring (inside diameter 20mm and 1.5mm thick). These were sitting on a thin polyethylene sheet backed by a flat glass plate. The molds were very lightly lubricated with Dow Corning High Vacuum Silicone Grease. Another flat glass plate faced with a sheet of polyethylene was used to cover the $Ca(OH)_2$. The assembly was held together by means of a "C" clamp and placed in constant temperature humidity chamber held at $37^{\circ}C$ and near 100% humidity.

After one hour, the specimens were withdrawn from the oven and two specimens placed in each tared weighing bottle and weighed. The combined weight of the specimens and the weighing bottle, less the weight of the weighing bottle were taken as the weight of the specimens of $Ca(OH)_2$. The specimens were submerged immediately by pouring 50ml of distilled water into the weighing bottles, which were stored for 23 hours at $37^{\circ}C$. There was to be no evidence of crystal growth on the surface of the specimens. The water was evaporated from the weighing bottle at temperature just below $100^{\circ}C$. The weighing bottle was then dried at $150^{\circ}C$ to constant weight. After cooling to room temperature in a dessiccator containing thoroughly dry anhydrous calcium sulfate (Drierite), the weighing bottles and contents were weighed with a precision of 0.2mg. This cycle of heating the weighing bottle to $150^{\circ}C$, cooling over a dessiccant, and reweighing was repeated until the weight loss of

the bottle was not more than 0.5mg in any 24 hour period. The difference between the final weight of the weighing bottle and its initial weight was taken as the amount of disintegration. The gain in weight divided by the weight of the specimen times 100 gave the percentage of disintegration. The average of 10 duplicate tests (ten weighing bottles containing two specimens each) were reported to nearest 0.1%.

Acid Solubility. Specimens prepared similarly to those for water solubility were used. Ten specimens from each brand of hard setting calcium hydroxide were used. The net weight of the specimens were obtained. The specimens were suspended by a stainless steel frame wire in a small beaker (50ml) so that they were immersed in 37 percent (by weight) orthophosphoric acid solution (H3PO4) for 60 seconds. Upon removal from a beaker the two exposed surfaces of the specimens were rinsed with a stream of tap water for one minute. The surfaces were then brushed lightly with a camel's hairbrush to remove loosely adhering debris and again rinsed with water. The specimens then were blotted dry with absorbant tissue and weighed. The difference between the initial weight and the weight after the specimens immersed in the acid was considered the weight loss of the specimens. The loss in weight divided by the weight of the specimen times 100 gave the percentage of acid solubility.

<u>Compressive Strength</u>. The specimens were prepared in a split brass mold with internal dimensions of 12mm high by 6mm wide. The mold was placed on a flat glass plate covered by a thin polyethylene

sheet and slightly overfilled with a portion of the mix of hard setting Ca(OH)₂ within three minutes after commencing the mixing. A second flat glass plate and polyethylene sheet was pressed on the top of the mold and held together with a "C" clamp. The molds were coated with a mold release agent (Miller-Stephenson Release Agent, ng-122). Three minutes after the start of the mix, the Ca(OH)₂ in the mold was transferred to a constant temperature water bath held at 37°C. One hour later the ends of the cylinder were surfaced plane at right angles to the axis using a 240 grit silicon carbide paper. The test specimens were kept wet during the grinding. After surfacing, the specimens were removed from the molds and kept in 100% relative humidity at 37°C. The time lapse between the start of the mixing and crushing was 24 hours. The specimens were loaded in compression at a head speed of 0.05 in/min on an Instron Universal Testing Machine using a 1000 pound load cell at 200 pounds full scale except for Life where full scale was set at 500 pounds. Force required to crush the specimens was recorded on a Houston Super Scribe 4900 strip chart recorder at 10 in/min.

The value for compressive strength was reported as the average of six or more from the lot of ten specimens. If the values for individual specimens fell more than 15% below the average of the ten, they were discarded and the average of the remaining specimens reported. If more than four of the specimens were eliminated, the test was repeated.

Bacteriocidal Test. The antibacterial effect of hard setting $Ca(OH)_2$ was evaluated by using Streptococcus mutans.

The hard setting $Ca(OH)_2$ was mixed according to the manufacturer's instructions. Two methods were used to conduct this study. A stainless steel mold with dimensions: 6mm wide by 3mm thick was used to fabricate specimens. A mold release agent (Dow Corning High Vacuum Silicone Grease) was used to permit removal of specimens. The molds were placed on a flat glass covered with a thin polyethylene sheet. Adequate quantity of $Ca(OH)_2$ mixed and placed in the mold. Another flat glass covered with polyethylene sheet was placed over the molds and transferred to a constant temperature humidity chamber held at $37^{\circ}C$ and near 100% humidity for one hour, then the specimens were removed from the molds.

After the mitis salivarius agar plates were prepared, Streptococcus mutans were spreaded over them. Calcium hydroxide specimens were placed on the plates and incubated in an atmosphere contained 10% CO_2 , 5% hydrogen and 85% nitrogen at 37°C. After 48 hours, the plates were inspected for microorganisms growth and inhibition zones size were compared.

The other method employed placement of freshly mixed materials directly into cavatus formed in the blood agar plates. Cavatus were prepared by use of a number three cork bores resulting in cavatus with an approximate diameter of 7mm. Pluges of agar were recovered from the blood agar plates formed by the cork borer by aspirating with Tygon tubing. The blood agar plates were incubated in a similar manner to that for mitis salivarius. After 48 hours the zone of inhibitions were photographed and compared.

<u>Hydroxyl Ions Diffusion</u>. Agar plates with phenolphthalein indicator were used to detect the amount of diffusion of OH^- in a manner similar to that used for antimicrobial activity, that is, the diameter of color change was measured and compared for each brand of hard setting $Ca(OH)_2$ material.

The hydroxyl ions diffusion plates were prepared by adding 7.5g agar (Difco Special Agar - Noble 0142-02) to 500cc water at 100° C while stirring on a magnetic hot plate. 10cc phenolphthalein (1% W/V in 95% alcohol, Ricca chemical # 5620) was added upon cooling to 45° C.

Cavatus were formed in the agar/phenolphthalein plates in a manner similar to that prepared for bacteriocidal studies. Freshly mixed $Ca(OH)_2$ material was placed in the cavatus and diffusion allowed to proceed at room temperature. The diameter of color change was measured after two to four hours and photographs of the plates were taken to compare the difference in diffusion

<u>Calcium Ions Diffusion</u>. Agar was prepared in a similar fashion to that for OH^- diffusion.

Upon cooling of the agar (500cc) to 45° C on a magnetic hot plate approximately 1.0g calmagite (1-(1- hydroxy -4- methyl -2phenylazo) -2- napthol -4- sulfonic acid; Mallinckrodt, Inc. 4283) was added using the supplied dispenser (10 dashes), along with 10cc buffer containing 57cc concentrated NH₄OH, 7.0g NH₄cl in 100cc water. The resultant pink color was titrated to a blue end point using saturated (10g/100cc water) EDTA disodium salt (Mallinckrodt, Inc. 4931) with a standard eye dropper (approximately 28 drops). Freshly mixed $Ca(OH)_2$ material was added to the agar plates in a similar fashion to that for OH^- diffusion. Then, the plates were photographed and compared for Ca^{++} diffusion.

CHAPTER IV

RESULTS

<u>Water Solubility</u>. The results for the water solubility and disintegration of the hard setting calcium hydroxide materials Dycal, Life, Procal and Renew are shown in Tables 2-5. The tables show the specimen number, the original weight of the specimens, the weight of the residue calcium hydroxide left after 23 hours in the weighing bottles, the amount of disintegration in percentage that was reported in the ten tests and the last column shows summary of the means and standard deviations of the samples.

The statistics for these tests are shown in Table 14, the mean and standard deviation for the percentage of solubility after 23 hours of each sample was as follows: Dycal was 2.73 ± 0.17 . Life was 5.29 ± 0.36 . Procal was 5.10 ± 0.19 and Renew 6.02 ± 0.5 . Dycal had the lowest amount of water solubility, and Renew the highest while Life and Procal fell in between the other two groups. The results are also demonstrated by the histiogram in Figure 1.

The results of tests on specimens of Life prepared with different base-catalyst ratios are present in Table 17. These data indicate that the water solubility of this product appears to be very sensitive to the relative amounts of the two pastes used for making the mix. When the ratio of base to catalyst paste was increased from 1:1 to 1.2:1, the

solubility of latter specimens was greater than those prepared with the lower base to catalyst ratio. [The values for Dycal were statistically different from Life, Procal and Renew as determined by T. statistics (P < .01)]

Acid Solubility. The results of acid solubility tests on specimens of hard setting calcium hydroxide Dycal, Life, Procal and Renew are presented in Tables 6-9. The means and standard deviations for these materials were as follows: Dycal was 3.96 ± 0.39 . Life was 0.89 ± 0.06 . Procal was 9.84 ± 0.64 and Renew 4.4 ± 0.34 . Life had the lowest amount of orthophosphoric acid solubility followed by Dycal, Renew, while Procal had the highest amount of acid solubility. Table 14 shows statistics, the mean and standard deviation, for the four materials. The acid solubility results are demonstrated by the histiogram in Figure 2. These data indicate that acid solubility did not follow the same manner for water solubility. Table 18 shows data for acid solubility of Procal, these results were different from the data shown in Table 8. [The values for Life were statistically significantly different from Dycal, Procal and Renew (P <.01). The values for Procal were significantly higher than those for Dycal, Life and Renew (P <.01)]

<u>Compressive Strength</u>. The results for the compressive strength tests of hard setting calcium hydroxide materials Dycal, Life, Procal and Renew are presented in Tables 10-13. The mean and standard deviation for the compressive strength are shown in Table 14.

The mean and standard deviation of each sample was as follows: Dycal was 2424 \pm 71 psi (19.4 \pm 0.6 MPa), Life was 4870 \pm 535 psi $(38.9 \pm 4.3 \text{ MPa})$, Procal was $2128 \pm 78 \text{ psi} (16.9 \pm 0.7 \text{ MPa})$ and Renew was $1879 \pm 67 \text{ psi} (15 \pm 0.5 \text{ MPa})$.

The compressive strength results are also demonstrated by the histiogram in Figure 3, with the strength (psi) shown in the left hand vertical column. Life outperformed the other three base materials 2 to 1. [The values for Life were statistically significantly different from Dycal, Procal and Renew (P < .01)]

Bacteriostatic Effect. The bacteriostatic tests results after 48 hour incubation period of Streptococcus mutans in mitis Salivarius agar and blood agar media are shown in Figures 4 and 5. These tests were qualitative and indicated that Procal had the most powerful inhibitory effect on the micro organisms followed by Dycal, Renew and Life. The micro organisms grow directly into the calcium hydroxide materials in case of Life $Ca(OH)_2$ plates.

<u>Hydroxyl Ion Diffusion</u>. The results for the tests of hydroxyl ion diffusion are demonstrated in Table 15. The materials listed in their order of decreasing distance of hydroxyl ion diffusion measured in cm are Dycal (1.6 \pm 0.4 cm), Life (1.6 \pm 0.2 cm), Renew (1.5 \pm 0.2 cm), Procal (1.3 \pm 0.1 cm), Hydrex (0.9 \pm 0.1 cm) and MPC (0.8 \pm 0.3 cm).

The diffusion was measured at four, three and two hours and the plates were photographed after five hours. The hydroxyl ions diffusion are shown in Figure 6.

<u>Calcium Ion Diffusion</u>. The calcium ion diffusion results are presented in Table 16. The materials were measured at four, three and two hours. The materials listed in their order of decreasing distance of calcium ino diffusion are Dycal $(2.5 \pm 0.2 \text{ cm})$, Renew $(2.4 \pm 0.1 \text{ cm})$, Procal $(2.3 \pm 0.1 \text{ cm})$, Life $(1.8 \pm 0.1 \text{ cm})$, Hydrex $(1 \pm 0.1 \text{ cm})$ and MPC $(0.9 \pm 0.3 \text{ cm})$. The measurements at four, three and two hours are parallel to each other. The calcium ions diffusion are shown in Figures 7 and 8.

Water Solubility of Hard Setting $Ca(OH)_2$ Bases

	Brand: Dycal					
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Summary x±s (%)		
1	1210.3	32	2.64			
2	1215.1	35.6	2.92	0 72 0 17		
3	1217.9	33.4	2.74	2.73±0.17		
4	1186.7	33.8	2.84			
5	1180.9	27.8	2.35			
6	1175	35	2.97			
7	1191.7	32.7	2.74			
8	1339.8	35.5	2.64			
9	1228	34.9	2.84			
10	1195.1	31.9	2.66			

Water Solubility of Hard Setting Ca(OH)₂ Bases

<u> </u>	Brand: Life					
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Summary X±s (%)		
1	1174.1	57.9	5.02			
2	1174.1	59.5	5.06	5 20 10 20		
3	1153.3	56.1	4.86	5.29±0.36		
4	1159.3	59.2	5,10			
5	1150.3	55.3	4.80			
6	1146.6	62	5.40			
7	1147	65	5.66			
8	1131.5	62.7	5.54			
9	1153.2	66	5.72			
10	1178.8	68	5.76			
	·					

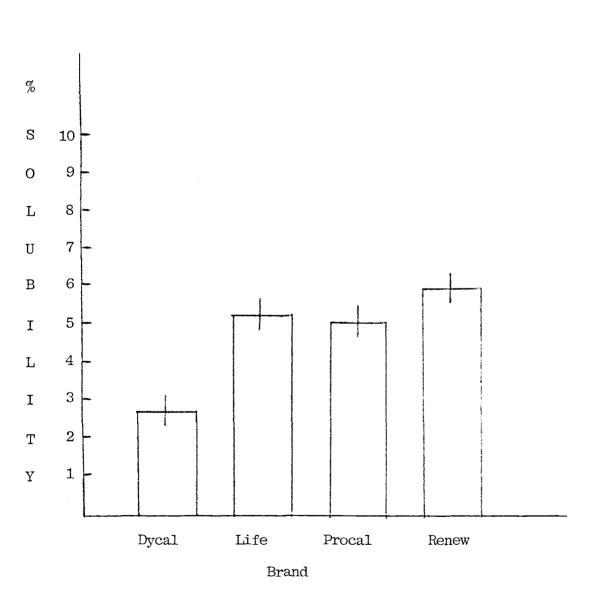
Water Solubility of Hard Setting $Ca(OH)_2$ Bases

	Br	and: Procal	· · · · · · · · · · · · · · · · · · ·	
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Summary X±s (%)
1	1220	59	4.83	
2	1223.6	60.6	4.95	5.10±0.19
3	1232.8	66.3	5.37	
4	1256	62	4.93	
5	1240.4	65.5	5.28	
6	1221.8	60.5	4.95	
7	1238.3	65.5	5.28	
8	1230.5	65	5,28	
9	1251.8	63	5.03	
10	1237.8	64.3	5.19	

Water Solubility of Hard Setting $Ca(OH)_2$ Bases

	Brand: Renew					
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Summary x±s (%)		
1	1320	86.2	6.53			
2	1291.5	68.4	5.29	6.02±0.50		
3	1321.7	89.7	6.78			
4	1331.3	76.4	5.73			
5	1320.5	83.7	6.33			
6	1313.5	78.9	6			
7	1308.6	82.4	6.29			
8	1340	83	6.34			
9	1297.2	70.4	5.42			
10	1325	73.2	5.52			

Figure 1



Water Solubility of Hard Setting $Ca(OH)_2$ Bases

Acid Solubility (H_3PO_4 , 37%) of Hard Setting Ca(OH)₂ Bases

	Brand: Dycal				
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Summary x±s (%)	
1	606.6	22.6	3.72		
2	601.2	27.5	4.57		
3	599.4	24.2	4.03	3.96±0.39	
4	588.2	20.6	3.50		
5	590.8	25.6	4.33		
6	606	21.5	3.54		
7	587.8	25.3	4.30		
8	600.5	25.7	4.27		
9	604.4	21.2	3.50		
10	611.5	23.7	3.87		

Acid Solubility (H_3PO4, 37%) of Hard Setting $Ca(OH)_2$ Bases

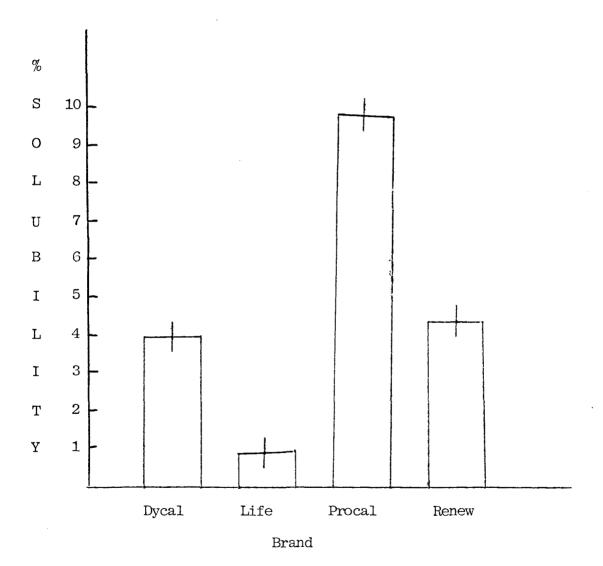
	Brand: Life				
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Summary X±s (%)	
1	587.3	5.7	0.97		
2	574.2	5.5	0.95	0.89±0.06	
3	578	5.2	0.89		
4	573	4.8	0.83		
5	570.3	4.6	0.80		
6	593.4	5.2	0.87		
7	573.8	5.7	0.99		
8	565.9	4.9	0.86		
9	596.5	5.3	0.88		
10	583.7	5.3	0.90		

Acid Solubility (H_3PO₄, 37%) of Hard Setting $Ca(OH)_2$ Bases

	Brand: Procal					
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Summary x±s (%)		
1	613.4	56.1	9.14			
2	629.5	56.2	8.92	9.84±0.64		
3	610	63.2	10.36			
4	581.1	56.2	9.67			
5	633.7	62	9.78			
6	635.2	59.5	9.36			
7	603.7	59.4	9.83			
8	603.5	60.7	9.99			
9	620.5	67	10.79			
10	615.7	66.5	10.80			

Acid Solubility (H_3PO_4, 37%) of Hard Setting ${\rm Ca}({\rm OH})_2$ Bases

	Brand: Renew					
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Sunmary x±s (%)		
1	665.7	32.1	4.82	4.4.0.24		
2	660.1	26.8	4.05	4.4±0.34		
3	672.3	27.4	4.07			
4	659.2	27.4	4.15			
5	652.3	30	4.59			
6	659.5	27	4.09			
7	647.3	26.7	4.12			
8	653.7	31	4.74			
9	640.9	29	4.52			
10	670.6	32	4.89			
	<u> </u>					



Acid Solubility of Hard Setting $Ca(OH)_2$ Bases

Compressive Strength of Hard Setting $Ca(OH)_2$ Bases

	Brand	: Dyc	al	
Specimen No.	Load at Failure (lbs)			sive Strength i (MPa)
				Mean_Summary x±s
1	102	2334	(18.7)	2424±71
2	107.8	2467	(19.7)	(19.4±0.6)
3	111	2540	(20.3)	
4	107.4	2458	(19.7)	
5	106	2426	(19.4)	
6	108.4	2482	(19.9)	
7	103.2	2362	(18.9)	
8	105	2417	(19.3)	
9				
10	102	2334	(18.7)	

Compressive Strength of Hard Setting $Ca(OH)_2$ Bases

	Brand	d: Li	fe	
Specimen No.	Load at Failure (lbs)			ssive Strength si (MPa)
	······································			Mean Summary X±s
1	184.2	4215	(33.7)	4970+525
2	204	4668	(37.3)	4870±535 (38.9±4.3)
3	186	4256	(34)	(00.011.0)
4	233	5332	(42.7)	
5	214.5	4908	(39.3)	
6	237	5423	(43.4)	
7	195	4462	(35.7)	
8	257	5881	(47)	
9	207.5	4748	(38)	
10	210	4806	(38)	

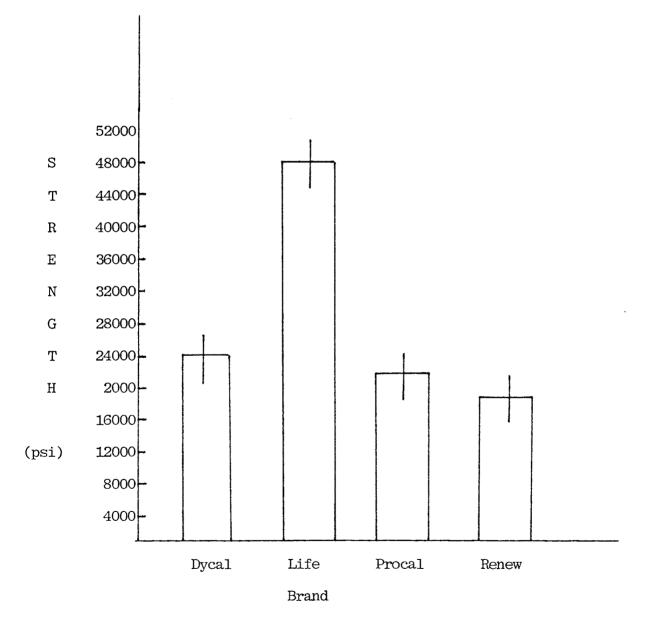
Compressive Strength of Hard Setting $\mbox{Ca}(\mbox{OH})_2$ Bases

	Brand	Procal	
Specimens No.	Load at Failure (lbs)	Comr	pressive Strength psi (MPa)
			Mean_Summary X±s
1	95.4	2183 (17.5)	2128±78
2	93.4	2137 (17.1)	
3	95.6	2188 (17.5)	(16.9±0.70)
4	92.8	2124 (17)	
5	93	2128 (17)	
6	89.8	2055 (16)	
7	88.6	2027 (16)	
8	100	2288 (18)	
9	91.6	2096 (16.8)	
10	89.8	2055 (16)	
	:		

Compressive Strength of Hard Setting $Ca(OH)_2$ Bases

Brand: Renew				
Specimen No.	Load at Failure (lbs)	Compressive Strength psi (MPa)		
				Mean Summary x±s
1	80.8	1849	(14.8)	1879±67
2	80	1830	(14.6)	
3	80.4	1840	(14.7)	(15±0.5)
4	80.6	1844	(14.8)	
5	80	1831	(14.6)	
6	86.6	1982	(15.9)	
7	87.6	2005	(16)	
8	80.5	1842	(14.7)	
9	84.4	1931	(14.7)	
10	80.2	1835	(14.7)	





Compressive Strength of Hard Setting Ca(OH)₂ Bases

Properties of Hard Setting $Ca(OH)_2$ Bases

	Brand				
	Dycal	Life	Procal	Renew	
Water Solubility Percentage Weight loss x±s (N=10)	2.7±0.2	5.3±0.4	5.1±0.2	6 ±0.5	
Acid Solubility Percentage Weight loss x±s (N=10)	4±0.4	0.9±0.1	9.9±0.6	4.4 ±0.3	
Compressive Strength PSI (MPa) x±s {N}	2424±70.5 (19.4±0.6) {9}	4870±534.8 (39_±4.3) {10}	2128±77.6 (17 ±0.6) {10}		

Time (Hrs.)	Brand Distance - cm $(\bar{x}\pm s)$					
	Dycal	Life	Procal	Renew	Hydrex	MPC
2	1.4±0.1	.9±0.1	.9±0.4	1±0.4	0.5±0.1	0.4±0.1
3	1.5±0.1	1.2±0.4	1.1±0.1	1.3±0.2	0.6±0.1	0.5±0.0
4	1.6±0.4	1.6±0.2	1.3±0.1	1.5±0.2	0.9±0.1	0.8±0.3

Hydroxyl Ion Diffusion of Hard Setting $Ca(OH)_2$ Bases

Calcium Ion Diffusion of Hard Setting Ca(OH)2 Bases

Time (Hrs.)	Brand Distance - cm (X±s)						
	Dycal Life Procal Renew Hydrex MPC						
2	1.7 ± 0.2	1.3±0.1	1.6±0.1	1.6±0.1	0.7±0.1	0.4±0.2	
3	2±0.1	1.6±0.2	2±0.1	2±0.1	0.8±0.1	0.5±0.1	
4	2,5±0.2	1.8±0.1	2.3±0.1	2.4±0.1	1±0.1	0.9±0.3	

Water Solubility of Hard Setting Ca(OH) O_2 Bases

<u></u>	Brand: Life						
[®] pecimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percenmtage WeightJt Loss	Summary X±s (%)			
1	1117.3	85	7.6550	0.01 - 50			
2	1133.2	87.8	7.774	6.91±.58			
3	1174.3	81.5	6.9994				
4	1168.7	77.1	6.5009				
5	1138.6	86.6	7.6830				
6	1184.4	78.3	6.6830				
7	1140.8	76.6	6.771				
8	1164	73.9	6.3834				
9	1150	69.2	6.0001				
10	1165.2	82	7.0003				

Acid Solubility of Hard Setting $Ca(OH)_2$ Bases

	I	Brand: Procal	-	•
Specimen No.	Original Weight (mg.)	Weight Loss (mg.)	Percentage Weight Loss	Summary x±s (%)
1	618.2	66.9	10.82	10,001,05
2	628.5	61.8	9.83	10.66±.95
3	600.8	70.1	11.66	
4	626.6	56.7	9.04	
5	600.4	62	10.32	
6	624.9	68.9	11.02	
7	614.9	76.8	12.48	
8	609.2	66.4	10.89	
9	611.7	63.2	10.33	
10	618.2	63.2	10.22	

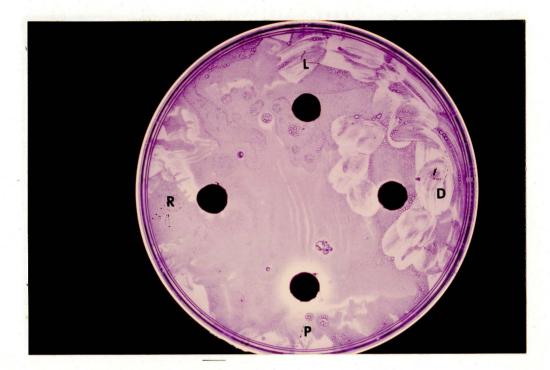


Fig. 4. Bacteriostatic effects of hard setting calcium hydroxide. D-Dycal, L-Life, P-Procal, R-Renew. Effect was noticed surrounding Procal and to a very slight extent surrounding Life.

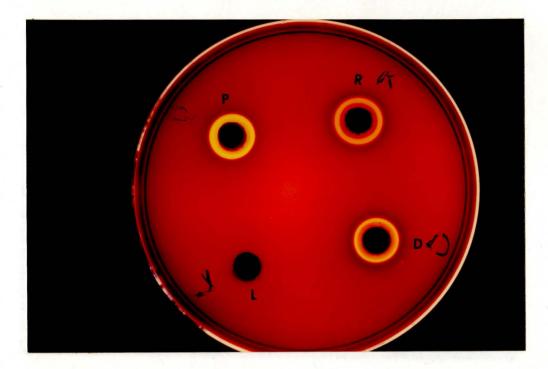


Fig. 5. Bacteriostatic effects of hard setting calcium hydroxide. Notice the clear halo surrounding Procal-P, double halos surrounding Dycal-D, and Renew-R. Very slight effect surrounding Life-L.

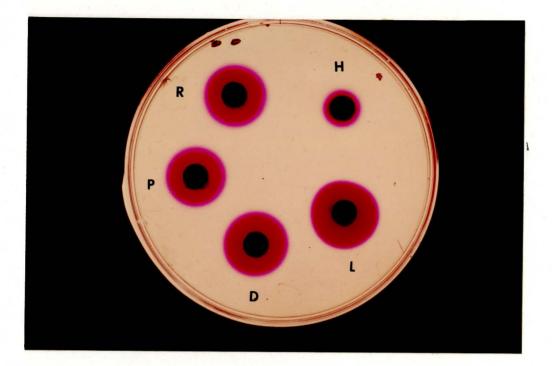


Fig. 6. Hydroxyl Ion diffusion of hard setting calcium hydroxide. Notice the difference in the distance of diffusion surrounding each brand. D-Dycal, L-Life, P-Procal, R-Renew, H-Hydrex.

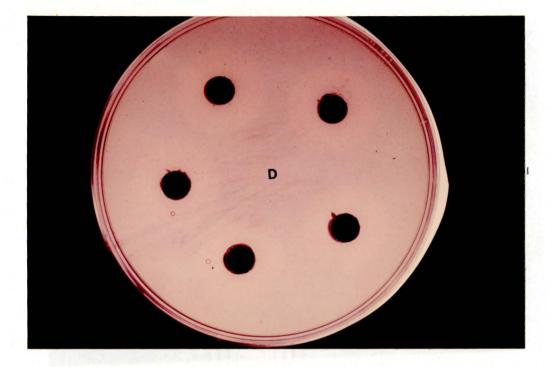


Fig. 7. Calcium Ion diffusion of hard setting calcium hydroxide. D-Dycal. Notice that the distance of diffusion are the same surrounding Dycal.

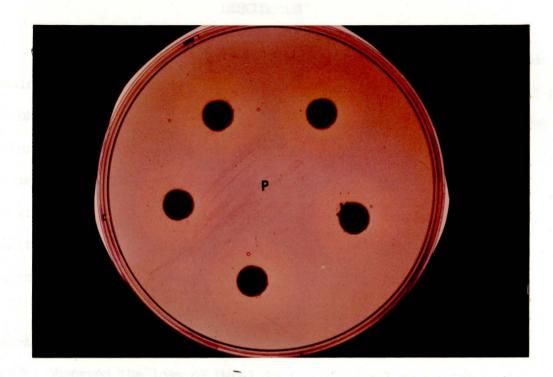


Fig. 8. Calcium ion diffusion of hard setting $Ca(OH)_2$. P-Procal. Notice the equal distance of diffusion surrounding Procal.

CHAPTER V

DISCUSSION

<u>Water Solubility</u>. Tables 2-5 show the actual amount of dissolved residue left in the weighing bottles after 23 hours, and the percent of solubility of the four brands tested. Dycal had the lowest value for water solubility, Renew had the highest, while Procal and Life fell in between the other two groups. Swartz (unpublished data) stated that it is likely that solubility in water would follow the same manner as solubility in acid. The present study indicates that is not likely the case, water solubility is totally different from acid solubility.

Solubility is a main factor which appears to favor failure of a calcium hydroxide base under amalgam restorations. Barnes and Kidd (1979) observed the loss of Dycal in four clinical cases. They indicated that of particular significance must be the clinical problem of distinguishing radiographically between a loss of a base and recurrence of caries.

The solubility of hard setting $Ca(OH)_2$ bases is used as a relative measure of the resistance of the base to the effect of oral fluids. This property is of particular importance for the base is relied upon to fill the space between the tooth and restoration. Solubility testing, as determined in the laboratory, has been carried out in distilled water rather than in natural or artificial saliva. The values for specimens immersed in saliva and water were very near to each other (Paffenbarger

1933) and distilled water has been used as a medium for storing cements. These 23 hour values in distilled water shown in Tables 2-5 are only a guide to clinical behavior since solubility in saliva under varied dietary conditions may be quite different from those laboratory tests.

The main reason for hard setting calcium hydroxide deterioration is that it is very soluble and rapidly deteriorates when exposed to aqueous solutions. Norman (1957 and 1959) indicated that this is in marked contrast to zinc phosphate and zinc polycarboxylate cements which show only slight surface deterioration after long periods of contact with aqueous solutions.

<u>Acid Solubility</u>. The data presented here indicates that there is a significant difference in the resistance of certain commercial hard setting calcium hydroxide preparations to attack by (37%) orthophosphoric acid solutions. Life was much less soluble than Dycal, Renew and Procal. Procal seems to have the highest value for acid solubility as shown in Table 8. These results are parallel with another study, Swartz (unpublished data).

Swartz suggested that some of these materials exhibited considerable deviation in solubility from specimen to specimen when the two components were visually proportioned by dispensing equal lengths of paste. Additional tests with Dycal showed that the acid solubility of this product was very sensitive to the base-catalyst ratio. The present investigation showed the same sensitivity of Life to base-catalyst ratio in water solubility tests, and Procal gave the same deviation in acid solubility. Norman and Associates have shown that the solubility of phosphate cements in organic acids, such as citric acid, is ten to twenty times greater than in distilled water; furthermore long term solubility may be much greater than indicated by the 24 hour test. Swartz et al. (1966 and 1968) and Eriksen (1974) found that Dycal proved to be quite impermeable to phosphoric acid leached from silicate and phosphate cement restorations. However, the ability of a calcium hydroxide material to prevent penetration of acids may be due to combination of acid neutralization and low permeability.

Observation of the four calcium hydroxide base materials during water and acid solubility shows that hard setting calcium hydroxide had high solubility in aqueous as well as in acid solutions. This is in contrast to other investigation (Trowbridge 1982) which indicated that failure of calcium hydroxide to significantly affect nerve impulse activity can probably be attributed to its low solubility. Caution should be exercised in its use and in the amount of material placed under a restoration.

<u>Compressive Strength</u>. The compressive strength tests of the four brands of the hard setting $Ca(OH)_2$ were carried out in a similar fashion described in the American Dental Association specification No. 8 and 9. The specimens were stored at 37°C and at a relative humidity of 100% for a period of 24 hours prior to crushing. The strengths attained by specimens under the laboratory conditions described here are not necessarily the values that are achieved by these materials at these specific times under oral condition. The compressive strength is useful for comparing materials which are brittle and generally weak in tension. Calcium hydroxide bases are brittle materials and are well suited to testing in compression. However, it is questionable whether a $Ca(OH)_2$ base ever fails as a result of pure compression, and it is more likely that the failure of the $Ca(OH)_2$ bases arises as the result of a complexity of forces.

Chong (1967) has shown that calcium hydroxide bases placed on firm dentin were not displaced. He indicated that a minimum compressive strength of 170 psi (1.2 MPa) is necessary to prevent displacement of the base during condensation of amalgam. While only a low strength is required to avoid the displacement of bases which are well supported on dentin, a considerably greater strength is essential when capping an exposed pulp (a situation for which calcium hydroxide is often recommended and where support is limited) if fracture is to be avoided. The present study indicates that all the four materials tested had a compressive strength greater than the values required to withstand displacement mentioned by Chong. Chong study showed that there was no difference in the compressive strength value of calcium hydroxide base after 7 minutes and 24 hours. This fact is very important because most of the restorations would be placed within 7 minutes after starting to mix the base.

In the previous investigation the calcium hydroxide materials were immersed in water for 24 hours or one week before they were tested, a situation which far removed from the actual practice. In this study the materials were not immersed in water; instead they were stored in 100% relative humidity at 37°C for 24 hours before crushing. This would prevent any effect the water might have on the compressive strength values of the hard setting calcium hydroxide.

The present study showed that the compressive strength for Dycal was 2424 psi, this result agrees with another investigation (Bryant, 1976) which indicated that the compressive strength for Dycal was 1919 psi. Bryant concluded that clinical consistencies of zinc phosphate cements, unmodified zinc oxide-eugenol cement, modified zinc oxideeugenol cements and calcium hydroxide cements will possess sufficient early strength to be used successfully beneath amalgam restorations.

Powers (1976) and Fuller (1980) indicated that the modulus of a base material is more important than the compressive strength and the ability of a base to support fillings depends on its modulus of elasticity. Powers found that the calcium hydroxide liner (Dycal) had higher values of modulus of elasticity and strength than the unmodified ZOE liner. Fuller added that the placement of zinc phosphate cement over calcium hydroxide bases did not reinforce the underlying Dycal.

Farah (1981) stated that knowing the modulus of elasticity of cement base enables one to determine its estimated failure load as a function of thickness. Farah indicated that the modulus of elasticity of zinc oxide-eugenol preparations that are not reinforced is approximately half that of calcium hydroxide. He suggested that the thickness of a base with a low modulus of elasticity, such as calcium hydroxide, should not exceed 0.5 mm.

The present study indicates that Life possessed the highest value

for compressive strength (4870 psi) followed; in order, by Dycal, Procal and Renew. Life is highly recommended under amalgam restorations and under stress bearing areas. Life also showed the lowest values for acid solubility, so it is highly indicated in acid etching technique especially as a base material under composite restorations in the posterior teeth.

<u>Bacteriostatic Effect</u>. The next property to be discussed is the bacteriostatic effect of hard setting calcium hydroxide. Procal proved to be the most efficient material in inhibiting Streptococcus mutan growth in the two types of media (mitis salivarius agar and blood agar). The other materials listed in their order of decreasing potency are Dycal, Renew and Life. These results are in accordance with other studies (Fisher 1977 and 1978). He indicated that Dycal had greater antibacterial effect than Hydrex. Fisher added that Procal would behave in a similar fashion to Dycal when used as cavity base material.

Fisher (1978) stated that the difference in the antibacterial capability between Dycal and Hydrex must be due to the difference in the ability of the calcium hydroxide to gain egress from the set material and produce an alkaline environment in the surrounding area. The pH measurements for Dycal was 11.5 and for Hydrex was 7 (Fisher 1977).

McWalter (1973, 1976) found that Dycal was more effective as a pulp capping agent than polycarboxylate cement and Keflin (an antibiotic). Forsten (1978) suggested that $Ca(OH)_2$ solution had an inhibitory effect on the microbial enzyme activity, probably due to its high pH. Mjor (1977) presented that bacteria cannot be demonstrated subjacent to $Ca(OH)_2$ base material. Fairbourn indicated that Dycal caused a

significant decrease in bacteria in deep carious lesions when placed in contact with carious dentin for five months. He added that re-entry to remove the residual carious dentin after indirect pulp capping with Dycal may be unnecessary, if the final restoration maintains its seal. Martin (1977) suggested that calcium hydroxide provides temporary root canal filling and may be used as a shorter term antiseptic canal dressing as an alternative to one of the more familiar canal medicaments, such as camphorated monochlorophenol. The material appears to have a beneficial drying action on persistent fluid exudates when used in this manner.

From the present study the exact mechanisms by which calcium hydroxide exerts its beneficial effects are poorly understood. Furthermore there was a wide variation among hard setting calcium hydroxide materials in their ability to inhibit bacterial growth, in vitro test system.

The size of the zone of bacterial growth inhibition does not necessarily reflect the strength of the antibacterial agent. The zone size may be influenced by the molecular size of the chemical and its diffusion constant. An agent that diffuses more easily will give a large zone. The fact that there was no inhibition zone for Life may mean only that it did not adequately diffuse through the medium.

The likely explanation for the larger zones obtained with the Procal material was that more free Procal was available for diffusion and not that it was more antibacterial.

The present investigations indicate that Life came second to Dycal in relation to hydroxyl and calcium ions diffusion when the two products were tested and calmagite or phenolphthalein was used as an indicator. The fact that there was no inhibition zone for Life may only mean that the pH around Life was lower than the other brands. Therefore, it is also conceivable that the lower pH was caused as consequence of a reaction between CO_2 (exposed to the material during incubation) and Life and the formation of insoluble material which did not adequately diffuse through the media.

As a wider appreciation of the clinical value of the calcium hydroxide material develops, there is need for further investigation into the biological basis for its action.

<u>Hydroxyl Ions Diffusion</u>. The hydroxyl ions diffusion results showed that Dycal had the largest zone of diffusion followed by Life, Renew, Procal, Hydrex and MPC. The hydroxyl ions diffusion has a direct effect on the mechanism of secondary dentin formation when calcium hydroxide used as a direct or indirect pulp capping material. Schroder (1971) suggested that the concentration of the hydroxyl ion rather than the calcium hydroxide per se is decisive in the tissue changes and secondary dentin formation.

Calcium hydroxide is very alkaline, pH about 12. When applied to a loose connective tissue it has a caustic effect, to which the underlying tissue responds with bordering tissue formation. Mitchell (1958) claimed that calcium hydroxide implanted subcutaneously in the connective tissue in rats induces heterotopic bone formation. Major carried out similar experiments on the rat, but concluded that he could not demonstrate any osteogenic potential of the calcium hydroxide.

Holland (1979) showed the development of complete hard tissue

bridges, that protect and keep the pulps free of inflammatory reaction, occurred in nearly all the cases when pulpotomy with use of calcium hydroxide is done. He added that on cavities restored with zinc phosphate and silicate cement, the description of inflammatory reactions in the presence of these materials is common.

<u>Calcium Ions Diffusion</u>. The calcium ion diffusion results are presented in Table 16. Dycal had the largest distance of diffusion. The other materials arranged in their decreasing order of diffusion are Renew, Procal, Life, Hydrex and MPC.

An explanation for the small diffusion of Hydrex and MPC can be offerred in the light of their structure. Hydrex and MPC when set, consist essentially of the calcium salicylate chelate and excess calcium hydroxide in a matrix of a paraffin type solvent. The other products such as Dycal are very similar except that, in place of paraffin, ethyl toluene sulphonamide is present as a plasticizer. The paraffin matrix of Hydrex and MPC is hydrophobic in nature and probably prevents the diffusion of water into the set material, whereas the ethyl toluene sulphonamide of Dycal, has polar groups and will allow diffusion of water into the material. The egress of calcium hydroxide results from producing an alkaline environment in the surrounding area. This important factor should be taken into account in any future developments of materials of this type.

The calcium ion diffusion results appear to be slightly different from that for hydroxyl ions diffusion but Dycal had the highest value of diffusion in both of them. Phenolphthalein was used as hydroxyl ions

indicator while calmagite used as calcium ions indicator.

Torneck (1980) indicated that calcium hydroxide base has a stimulating effect on the repairing pulp and promotes cell mitosis. This stimulating effect may be associated with the local tissue pH and the presence of calcium ions, which disassociate from the calcium hydroxide and diffuse through the dentinal tubules, coming in contact with the pulp. Sciaky (1960) proposed that the calcium ions from the calcium hydroxide do not enter into the formation of the new dentin bridges. The calcium icons necessary for the calcification of new dentin are therefore probably derived from the pulp itself. Attala (1969) examined the incorporation of calcium into the pulp by radioactive techniques. Results of the study indicated a diffusion of calcium into the pulp, but that it did not participate in the formation of dentin.

The differences in the patterns of release of calcium could be explained on the basis of the composition of the products. The differences of opinion on the possible sources of calcium in the formation of reparative dentin may be a result of such composition considerations.

The present study results compare favorably with previous studies. Dycal material produced the greatest calcium ions diffusion while Hydrex and MPC produced the least. A clinical study (Fisher 1977) found that Dycal was more effective than Hydrex in sterilizing dentin.

A high release of calcium and high alkalinity appear to be clinically advantageous. A high rate of release of hydroxyl and calcium ions necessary to sterilize and remineralize carious dentin, even if the base eventually disintegrates. Analysis of the present in vitro data indicates that these bases of hard setting calcium hydroxide will be rapidly washed away in service. Even when in comparatively protected positions at the base of a cavity, they will be exposed to water which has diffused along the dentinal tubules. Clinical observations indicate that Dycal does indeed disappear from cavity bases over which amalgam restorations have been placed (Barnes 1979).

It is clear that most of the <u>in vitro</u> and clinical studies were conducted with Dycal, whether the other three brands of $Ca(OH)_2$ behave similarly to Dycal needs to be verified with further clinical and laboratory studies. The present study indicates that Life had the highest compressive strength and lowest acid solubility values. It is appropriate to suggest that Life is highly recommended in acid etching technique and under amalgam restorations, followed, in order, by Dycal, Renew, Procal.

All of the $Ca(OH)_2$ bases are affected by water to varying degrees, and the bases must be considered to be hydrolytically unstable. The compositions of the material play as essential role in controlling the rate of disintegration of the bases. The compositions of Hydrex and MPC limit the aqueous attack upon the hydrolytically unstable matrix and the excess calcium hydroxide. By contrast, the hydrophilic nature of composition in Dycal, Life, Procal and Renew allows the free passage of water, which then attacks the vulnerable cement structure, leading to complete disintegration of these bases. Decomposition of the $Ca(OH)_2$ bases appears to be clinically advantageous, since the large amount of calcium released at high alkalinity promote sterilization and calcification of carious dentin.

Calcium hydroxide has been the material of choice for use in pulp capping over healthy pulps to induce reparative dentin.

The mechanism for the induction of dentin formation and repair under calcium hydroxide may be disturbed when $Ca(OH)_2$ placed over an inflamed or cariously exposed teeth, a very common clinical situation. The following undersirable effects may occur as a consequence of a reaction between the inflamed pulps and the calcium hydroxide: complete root canal calcification, pulp necrosis and internal resorption.

Calcium hydroxide should not be used to treat existing pulpitis. It has no curative effect for inflammation. Caution must be exercised when $Ca(OH)_2$ used as a pulp capping material.

CHAPTER VI

SUMMARY AND CONCLUSION

In this study the following properties of hard setting calcium hydroxide base materials were investigated. First, the water and acid solubility was studied. Second, a number of parameters were determined, namely; compressive strength, diffusion of calcium and hydroxyl ions in an agar gel and bacteriostatic properties.

1. Dycal had the lowest values for water solubility and Renew the highest while Life and Procal fell in between the other two groups.

2. Life had the lowest values for 37% orthophosphoric acid solubility and Procal the highest. The acid solubility for the four brands did not follow the same order for water solubility.

3. The base-catalyst ratio affected the acid and water solubility of $Ca(OH)_2$.

4. The compressive strength value for Life are twice that of the other brands.

5. Hard setting calcium hydroxide bases are capable of diffusion and thus of expanding the area of antibacterial effect. Procal appears to have the greatest antibacterial effect on Streptococcus mutan by measuring the zone of growth inhibition on blood agar plates and mitis salivarius agar plates over time periods. Life showed no growth inhibition, which may be related to the lower pH surrounding the material.

6. Dycal and Life possessed the largest distance for hydroxyl and calcium ions diffusion and Hydrex and MPC showed the smallest distance of diffusion, while Procal and Renew fell in between the other two groups. The different properties of the materials can therefore be explained in terms of the hydrophilic nature of the organic matrix or carrier. In the case of Hydrex and MPC the matrix is oily, hydrophobic paraffin type of material whereas with Dycal the matrix is ethyl toluene sulphonamide which is polar and therefore less hydrophobic.

In conclusion, hard setting calcium hydroxide appears to be the most successful agent in inducing secondary dentin formation. Calcium ions deposited in the secondary dentin seem to come from the circulatory system. Recently, calcium hydroxide cements have become popular as alternatives to zinc oxide-eugenol cements, because of their ability to sterilize carious dentin and stimulate the formation of secondary dentin. However, these cements are hydrolytically unstable. The decomposition of the calcium hydroxide bases appears to be clinically advantageous, since the large amount of calcium released at high alkalinity promote sterilization and calcification of carious dentin. Whether the calcium hydroxide pH, its hydroxyl group or its Ca⁺⁺ group has the principle mechanism in the reparative dentin formation is not clear yet.

Hard setting calcium hydroxide appears to withstand forces required for amalgam condensation without displacement. Life had a relatively high compressive strength and low acid solubility, it is highly recommended in acid etching technique and under amalgam restorations. It would be advisable to see this study continue and clinical research done on water and acid solubility, long term solubility on these hard setting calcium hydroxide bases.

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APPROVAL SHEET

The thesis submitted by Mohamed S. Hwas has been read and approved by the following committee:

> Dr. James L. Sandrik, Director Professor, Dental Materials, Loyola

Dr. Douglas C. Bowman Professor, Physiology and Pharmacology, Loyola

Dr. Gary N. Taylor Associate Professor, Endodontics Loyola

Dr. Adalbert L. Vlazny Professor, Operative, Loyola

The final copies have been examined by the director of the thesis and the signature which appears below verifies the fact that any necessary changes have been incorporated and that the thesis is now given final approval by the Committee with reference to content and form.

The thesis is therefore accepted in partial fulfillment of the requirements for the degree of Master of Science in Oral Biology.

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