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## The in Vitro Effect of Zoe, Calcium Hydroxide, and Zinc Cement Dental Bases on the Hardness of Human Dentin

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THE IN VITRO EFFECT OF ZOE, CALCIUM HYDROXIDE, AND ZINC  
CEMENT DENTAL BASES ON THE HARDNESS OF HUMAN DENTIN

by

Michael S. Borzello, B.S., D.D.S.

A Thesis Submitted to the Faculty of the Graduate School  
of Loyola University of Chicago in Partial Fulfillment  
of the Requirements for the Degree of  
Master of Science

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## VITA

Michael S. Borzello was born in Chicago, Illinois on September 14, 1946.

After graduation from Proviso West High School, Hillside, Illinois, in 1964, Mr. Borzello attended Central Missouri State University in Warrensburg, Missouri. After completing college, he pursued a dental career at Loyola University School of Dentistry from 1969 to June of 1973 when he received the degree of Doctor of Dental Surgery.

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To Loyola University for allowing me to pursue my endeavors.

## DEDICATION

I dedicate this thesis to my friends and family,  
who have made my studies possible.

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## CHAPTER I

### INTRODUCTION

Over the years, many new techniques and materials have been introduced to dentistry. Dentistry would not be the precise discipline it is today if the technology present in our modern society did not exist. It behooves the practitioner of today to understand the materials and techniques that he has at his disposal so that he can intelligently decide how to treat his patients.

One of the types of materials that is in common use by practitioners are the dental bases. They are used for the protection of the pulp, or to aid the pulp in recovering from irritation. Sturdevant (1968) lists three criteria in selecting an intermediate base as follows:

1. the ability of the material to protect the pulp
2. the ability of the material to eliminate or prevent postoperative discomfort
3. the effect of the material on the clinical success of the restoration.

The total effect of the dental bases is a sum of all of their properties, such as compressive strength, pH, thermal conductivity, and pulpal sedation. One of the effects that dental bases have on the dentin is to change the hardness of the dentin.

An in vitro experiment designed to determine the direct effect of dental bases on the hardness of human dentin was carried out. Any effect seen would be due to a direct physical or chemical effect of the dental base. Once the dental practitioner is aware of the direct effects



and indirect effects of the materials that he uses, he can use the sum of his knowledge to treat his patients more precisely according to their individual needs.

The three most frequently used dental bases are zinc phosphate cement, calcium hydroxide, and zinc oxide and eugenol. These three materials have at various times been in great regard or in great disregard as various authors' research have claimed the materials to be either beneficial, benign, or damaging to the pulp. Since most dentists do not prepare mixtures of these materials from their basic ingredients, but buy a commercial preparation designed for ease of manipulation and convenience, well known brands of commercial preparations were tested.

## CHAPTER II

### REVIEW OF THE LITERATURE

Hodge and McKay (1933) used a Microcharacter Hardness Tester with motor drive and diamond cutter point developed by C. H. Bierbaum to measure the hardness of dentin and enamel. The machine applied the diamond point under a constant load to a constantly moving sample. A microcut was formed. The Bierbaum hardness number was derived from the ratio of the width of the cut by the force. The researchers found crown dentin slightly harder than the root dentin (Bierbaum numbers 130 and 140 respectively).

Hodge (1936) tested Brinell, Rockwell, Monotron, Shore Scleroscope, Herbert Pendulum, and Microcharacter hardness testers to see which was most suited for dental hardness measurements. He found that the Microcharacter Hardness Tester caused no destruction of specimens and exhibited sensitivity to the different hardness of the dentin and enamel while the others did not.

Totah (1942) determined the effect of polishing and drying on the hardness of human dentin by the microcut method developed by C. H. Bierbaum. The extracted teeth were sectioned with a circular steel saw, mounted in plasticine, and tested. The teeth and sections were stored in water. Some sections were polished with emery paper and final polished with a moistened abrasive powder on a buffer wheel. The polished sections had a hardness of 140 Bierbaum. The unpolished sections were tested and had a hardness of 143 Bierbaum. The polished sections were

then dried for 44 hours in a 105°C oven. The dried, polished sections had a hardness value of 217 Bierbaum. He concluded that polishing has a minimal effect on the hardness of dentin but drying increases the hardness markedly.

Craig and Peyton (1958) determined the hardness of enamel and dentin with a Knoop diamond indenter mounted on a MO Tukon microhardness tester. They tested mature, freshly extracted, non-carious teeth. The teeth were embedded in plastic, sectioned with a water cooled wheel, polished by the successive use of 240A, 400A, and 600A Norton Tufback Speed wet paper supported by a glass slab, followed by polishings with Shamva and CRO metallographic polishes at low speeds on a wet polishing wheel. They tested loads from 25 to 200 grams and found that a 50 gram load applied for 15 seconds was optimum. An optimum load was then defined as a load that produced an indentation that had well defined borders with a minimum of fractured edges. They also found that dentin has a pronounced elastic recovery. They suggested that the measurements should be made within a few minutes of the indentation of the sample. They disregarded measurements that were outside twice the standard deviation of the average value. This came to approximately 15% of the total readings.

The authors found that dentin in the area of the dentinoenamel junction (DEJ) was the softest. They found the average for dentin was  $68 \pm 3$  KHN and the average for enamel was  $343 \pm 23$  KHN. They found no difference in hardness between root and crown dentin. They stated that their standard deviation for dentin was  $\pm 5$  KHN, which equaled approximately 7.5% error. They thought that this amount of error could account for an earlier study by Hodge and McKay (1933) that showed Bierbaum numbers 130 and 140

respectively for root and crown dentin.

Craig, et al, (1959) tested the Knoop microhardness of human dentin with a MO Tukon microhardness tester at loads from 2 to 25 grams for 15 seconds. The specimens were teeth selected and prepared as in their 1958 study. They found that loads of 10 grams and above showed less experimental variation than loads less than 10 grams. They also stated that a 10 gram load is more representative of the dentin's hardness than a larger load although they did not state how they arrived at this conclusion.

The authors found that the hardness of the crown dentin away from the DEJ and the pulp was the same hardness as root dentin away from the CEJ and pulp canal. Dentin near the CEJ, pulp canal, and DEJ was 15 to 30 KHN softer than the rest of the dentin. They also stained the sections with modified Pollak Trichrome stain. This staining procedure stained the organic material in the dentin darker than the inorganic material. Dentin near the CEJ, pulp canal, and DEJ stained darker. Dark staining and lower hardness value areas coincided. The difference in staining and hardness was attributed to the amount of organic material present.

Hegdahl and Hagebo (1972) evaluated load dependence in micro indentation hardness testing of enamel and dentin. They used a Vickers diamond pyramid indenter with the load applied for ten seconds. Maxillary first premolars with full root development, no caries, and no restorations were extracted. The teeth were stored in a buffered formalin solution. The teeth were wet sectioned with a Hamco Thin Sectioning Machine, embedded in plastic, polished on wet abrasive paper numbers 220, 320, 400, and 600 in succession. The final polish was accomplished using Buehler AB Alpha Polishing Alumina Number 2 on a

wet cloth.

The authors found that the hardness for enamel and dentin is load dependent. A change from 40 to 60 grams load increased the hardness from 319 to 322 in enamel. In dentin a load change from 15 to 30 grams changed the hardness value from 68 to 67. They found that less variation in measurement values resulted from increasing the load in the same specimens. Under 20 grams load the measurement variation increased rapidly.

Mjor, et al, (1961) investigated the in vivo effect of  $\text{Ca(OH)}_2$  and amalgam on 25 caries free teeth. Seventeen of the teeth were premolars slated for extraction for orthodontic considerations in children with an average age of 11.5 years. Seven were third molars and one was a supernumerary fourth molar extracted from young adults with an average age of 24 years. Preparations were made in the teeth, and the  $\text{Ca(OH)}_2$  or amalgam was placed. The  $\text{Ca(OH)}_2$  experimental teeth were then sealed with amalgam. Seven teeth were left unoperated to serve as a control group. The teeth were extracted from 15 to 139 days later. The teeth were embedded in plastic and wet sectioned with a Billings Hamco Thin Sectioning Machine. The sections were then tested with a Kentron Knoop Micro Hardness Tester with a 50 gram load applied for 15 seconds. They found that  $\text{Ca(OH)}_2$  caused a marked increase in the hardness of the dentin, and that amalgam effected little change in the hardness. They found the change in hardness apparent within as little as 15 days. Whether the effect of the  $\text{Ca(OH)}_2$  was pulpal or direct was not known.

Mjor (1962) investigated the in vivo effect of ZOE dental bases on the hardness of dentin. He measured the hardness with a Kentron Knoop Microhardness Tester. Nine caries free, newly erupted, first premolars, slated for

extraction for orthodontic considerations were used. Six of the teeth were in the experimental group and three teeth were used as a control. The teeth in the experimental group had an occlusal cavity prepared and ZOE placed in the floor of the preparation. The cavity was then sealed with amalgam. The teeth were extracted from 10 to 250 days later. They were embedded in plastic, wet sectioned with a Gillings Hamco Thin Sectioning Machine, and tested with a Kentron Micro Hardness Tester. They found that the ZOE covered dentin exhibited an increased hardness of 2.9 KHN. In his previous study, Mjor (1961) found that  $\text{Ca}(\text{OH})_2$  covered dentin in a similar experiment showed an increase of 9.8 KHN. The amalgam covered dentin in the 1961 study showed an increase of 0.3 KHN, which was not statistically significant.

Mjor (1967) in an in vivo experiment, made cavity preparations in 97 premolars slated for extraction for orthodontic considerations in children age 9 to 15 years of age. He then placed ZOE,  $\text{Ca}(\text{OH})_2$ , amalgam, and Ledermix (cortico-steroid/antibiotic mixture) over the exposed dentin. The preparations were then sealed with either amalgam or ZOE. The teeth were later extracted at various times from one-half hour to 117 days later. He found that there was no marked difference between ZOE and  $\text{Ca}(\text{OH})_2$  covered dentin stained with toluidine and alcian blue. He also found that ZOE treated dentin showed no change in microradiodensity, but that the  $\text{Ca}(\text{OH})_2$  treated dentin showed an increased microradiodensity.

Ripa, et al, (1972) studied the in vitro effect of  $\text{Ca}(\text{OH})_2$  and ZOE on the dentin of 32 sound, non-carious, unrestored, extracted premolars and molars. The enamel was removed from the occlusal surfaces of the teeth and ZOE or  $\text{Ca}(\text{OH})_2$  (Dycal) placed over one-half of the exposed

dentin. The untreated one-half served as the control. Sixteen teeth were treated with Dycal and sixteen teeth were treated with ZOE. The teeth were stored in a high humidity environment. Microradiographs were taken at two weeks, three months, six months, and one year. They found no difference in microradiodensity between control and ZOE-treated dentin and between control and  $\text{Ca}(\text{OH})_2$  treated dentin. They also studied the same materials in 32 carious, unrestored, extracted premolars and molars. Sixteen teeth were used for the ZOE sample and sixteen teeth were used in the  $\text{Ca}(\text{OH})_2$  sample. They were prepared and stored as previously described. Microradiographs were taken after two weeks, three months, six months, and one year. They found no increased microradiodensity in the ZOE treated carious dentin. They did find an increased microradiodensity in the  $\text{Ca}(\text{OH})_2$  treated dentin. They attributed this increase to penetration of  $\text{Ca}(\text{OH})_2$  into the carious dentin.

Ehrenreich (1968) tested the Knoop hardness of caries treated with  $\text{Ca}(\text{OH})_2$ , ZOE, and wax in vivo. He used a Kentron Microhardness Tester with a 100 gram load for 10 seconds. Teeth from children ages 8 to 9 years were used. Thirty-six asymptomatic unexposed teeth were divided into four groups of nine each. One group was a control group that had the cavity prepared with a high speed bur and spoon excavation. The teeth were then immediately extracted.

The experimental groups had the surface layers of decay removed with a high speed bur and spoon excavation as in the control group. The ZOE,  $\text{Ca}(\text{OH})_2$ , or wax was then placed on the floor of the preparation and the preparation sealed with amalgam. After eight weeks, the experimental teeth were extracted. The teeth were

washed overnight with water. They were then dehydrated for four hours with alcohol washes. The specimens were soaked in acetone for one hour and embedded in plastic. They were then sectioned with a Hamco-Gillings Thin Sectioning Machine. The sections were then subjected to either hardness testing or histological examination. The author found that the microhardness of the ZOE treated carious dentin increased appreciably though it did not reach the hardness of sound, non-carious dentin. The wax and  $\text{Ca}(\text{OH})_2$  treated dentin did not appreciably increase in hardness. The hardness increase in the ZOE group was 57 KHN. The increased hardness of the ZOE treated dentin was attributed to stimulation of the pulp by the ZOE to recalcify the decayed dentin. The histological study of the dentin showed that the dentin appeared to be normal in structure beneath the decayed areas. The  $\text{Ca}(\text{OH})_2$  and wax group showed a slight but statistically significant increase in hardness.

Wolf, et al, (1973) tested the in vivo effect of a ZOE base with approximately 2% fluoride, in the form of  $\text{CaFPO}_3$ , and a ZOE base without  $\text{CaFPO}_3$  added. The seven caries free premolars selected were slated to be extracted for orthodontic reasons. Two preparations were made in each tooth. One was filled with the  $\text{CaFPO}_3$ -ZOE and the other preparation was filled with the plain ZOE. The preparations were sealed with amalgam. After four to six weeks, the teeth were extracted. Seventeen teeth were selected and prepared the same as previously described, but were immediately extracted and the experimental bases applied and sealed with amalgam and stored in physiologic saline for 16 to 35 days. In preparation for hardness testing, the teeth were embedded in plastic, sectioned, and polished. A 100 gram load was



utilized for Knoop microhardness testing. They found the hardness increased approximately the same amount in both in vivo and in vitro samples beneath the  $\text{CaFPO}_3$ -ZOE (2.8 KHN and 3.3 KHN respectively). The hardness beneath the plain ZOE and adjacent unaffected control dentin did not show a significant change (0.3 KHN and 0.5 KHN respectively). The authors attributed the increased hardness to the additional Ca and F ions present in the  $\text{CaFPO}_3$ -ZOE mixture as compared to the plain ZOE mixture.

Biven, et al, (1971) studied the effect of eugenol and eugenol containing root canal sealers on the hardness of the dentin of extracted human teeth. The teeth selected were a random sample of permanent teeth excluding mandibular central and lateral incisors. The teeth had mature, fully developed roots. The teeth were stored in normal saline solution while being prepared for testing. The apical portion of the roots were sectioned off the root with a water spray handpiece. The apical portions of the roots were embedded in plastic in a Buehler Specimen Mounting Press. The samples were polished on a Buehler Fine Grinding Apparatus with progressive use of wet emery polishing paper of successive grits and a final polish with Buehler levigated alumina slurry. The tooth served as its own control. Before and after measurements were taken with a Kentron microhardness tester with a Knoop diamond indenter for 15 seconds with a 10 gram load. The specimens were stored in an analytical oven at  $37^\circ\text{C}$  and 90% relative humidity for 3 weeks. Eugenol and three brands of root canal sealers containing eugenol were tested. Two root specimens were in the eugenol group. Three root specimens were in each of the commercial root canal sealer groups. Two root apices were used as another control group. These two

root sections were mounted, polished and stored for 3 weeks to determine the effect of the preparation procedures. They found the hardness in the untreated control sample did not change significantly. The hardness of the eugenol and root canal sealer samples became significantly harder. The eugenol treated dentin became 11 KHN harder. The three root canal treated samples became 3 to 9 KHN harder.

Garberoglio and Brannstrom (1976) used a scanning electron microscope to study human dentin. They studied 24 extracted premolars, 5 extracted molars, and 1 extracted incisor. Sixteen of the teeth were from individuals 8 to 25 years of age. Fourteen of the teeth were from individuals 40 to 60 years of age. They found that the number of tubules in the dentin varies depending on proximity to the pulp. The number of tubules near the pulp chamber was  $45,000/\text{mm}^2$  and their diameter was 2.5  $\mu\text{m}$ . The number of tubules in the middle area of the dentin was  $29,000/\text{mm}^2$  and their diameter was 1.2  $\mu\text{m}$ . The peripheral dentin values were  $20,000/\text{mm}^2$  and their diameter was 0.9  $\mu\text{m}$ . They found that the tubules were various diameters and may or may not have odontoblastic processes in them. They found tubule volume was computed to be 10% on the average for coronal dentin. The range was 4% near the enamel and 28% near the pulp.

Outhwaite, et al, (1976) evaluated the radioactive iodine permeability of dentin from freshly extracted third molars. They found that increasing the temperature of the environment from  $25^{\circ}$  to  $35^{\circ}$  doubled the radioactive iodine penetration rate. Dentin proximal to the pulp was more permeable than dentin proximal to the enamel. The permeability of the dentin samples showed a slight increase for the first two days post-extraction. After

the second day, no change in the permeability rate was observed for the next four weeks.

Rothberg and deShazer (1966) studied the chelating ability of eugenol and ZOE. Third permanent molars without caries were immediately stored in 4% formalin after extraction. The 37 teeth were wet sectioned on a Hamco Thin Sectioning Machine. Two sections from each crown were used. Eugenol was added to 36 sections. Water was added to 14 sections. After 30 weeks, the amount of calcium removed from the sections by the eugenol was 4.1 mg./100 ml. Von Kossa staining of the sections showed decalcification of the sections. ZOE mixes were placed on seven sections for six weeks. Von Kossa staining showed surface decalcification of the sections. The authors stated that eugenol in the active form of the eugenolate ( $C_{10}H_{11}O_2$ ) is the chelating agent formed during the setting process of the ZOE. The eugenolate chelates the calcium from the dentin causing decalcification of the dentin.

Molnar (1967) mixed 100 gram samples of 80% zinc oxide and 20% eugenol. He then extracted the free eugenol from the mixtures by immersing them in methanol. The percentage of eugenol that was extracted depended on the amount of time the mixture was allowed to set. Immediately after mixing, 92.5% of the eugenol could be extracted. When ZOE with accelerators added was tested, immediately after hardening 10.9% of the eugenol was extracted. Accelerated ZOE set for 6 weeks had 5.8% of the eugenol extracted.

Batchelor and Wilson (1969) studied six brands of ZOE mixed under various atmospheric conditions. The temperatures varied from 20°C to 27°C. The humidity varied from 50 to 65% relative humidity. All the mixes conformed

to the FDI specification for dental silicate cements. The authors found that increasing the humidity or the temperature of the atmospheric conditions at the time of mixing speeded the setting reaction. The increased rate of reaction of the ZOE was caused by the reaction of the  $H_2O$  (humidity) with the eugenol. The reaction forms the active eugenolate, which reacts with the zinc oxide, which is also activated by  $H_2O$  (humidity). The authors concluded that the setting reaction of the ZOE was enhanced by high humidity.

Van der Lehr (1970) stated that zinc phosphate cements should no longer be used as a dental base or luting agent because of its acidity, which leads to pulpal death.

Powers and Dennison (1974) reviewed dental cements. They stated that the principal ingredient of zinc phosphate cement powder is zinc oxide. Magnesium oxide is also present in the powder to aid in its manufacture. The liquid is a solution of phosphoric acid and is approximately 33% water to which aluminum and zinc have been added as a buffer. The set zinc phosphate cement is essentially a hydrated amorphous network of zinc phosphate that surrounds incompletely dissolved particles of zinc oxide.

Swartz, et al, (1966) studied the permeability of dentin and dental bases to the constituents of zinc phosphate and silicate cements. Extracted sound molars were embedded in plastic. The crowns were reduced to cylinders 6 mm. in diameter and 3 mm. high with a lathe. A cavity of 4 mm. in diameter and 1 mm. deep was made in the top surface of the cylinder. The cavity formed in the cylinder of tooth was filled with silicate or zinc phosphate cement prepared from a liquid labeled with  $P^{32}$  phosphoric acid. The teeth were stored in water with

the cement covered with wax to prevent leaching. The teeth were sectioned 1, 24, and 72 hours after placement of the cement. Labeled  $P^{32}$  from both silicate and zinc phosphate cements penetrated the dentin. Copalite, Dycal, and Cavitec all reduced the amount of  $P^{32}$  that penetrated.

Norman, et al, (1966) studied the direct pH determination of samples of set cements utilizing a microelectrode. External and internal pH readings were taken of zinc phosphate, silicate, silicophosphate, and copper cements at 10 and 30 minutes, 1, 2, 4, 12, 24, and 48 hours, and 7 and 28 days after mixing. Junction pH determinations at the junction between fresh dentin and cements were undertaken at room and  $37^{\circ}C$  temperatures at 100% relative humidity.

The different brands of cement were approximately the same pH. The authors found that only very thin mixes of cement had a markedly lower pH than average or thick mixes. The pH of all the mixes had stabilized after 48 hours at 6.1 for the thin mix and 6.7 for the standard and thick mixes. The initial pH of the thin mix was 3.3, while the initial pH of the standard mix was 4.4. The dentin-cement interface measurements were not statistically different from the previous values. The standard mix was the mix that met ADA Specification Number 8.

The Council on Dental Materials and Devices of the A.D.A. (1967) states that the portions of zinc phosphate cement should be mixed in the following order, 1/16, 1/16, 1/8, 1/4, 1/4, and 1/4. The first three portions should be mixed for 10 seconds, the next two portions should be mixed for 15 seconds, and the last portion should be mixed for 30 seconds. A total mixing time of 90 seconds is the result.

Servais, et al, (1971) studied the setting and storage of zinc phosphate cements in humid and dry conditions. They studied the cement with X-ray diffraction, scanning electron microscopy, and electron microprobe analysis. They found that during the initial setting process, the essential component formed is a noncrystalline, amorphous, glassy phosphate. The cement consists of ZnO particles surrounded in the noncrystalline phosphate matrix. As the cement dries, extensive pores occur throughout the amorphous phosphate, and the whole mass becomes porous. The noncrystalline component is stable under relatively dry conditions at less than 30% relative humidity. At higher than 30% relative humidity, the surface layer transforms to the crystalline form, hopeite. Hopeite is  $Zn(PO_4)_2 \cdot 4H_2O$ .

Cartz, et al, (1972) placed freshly mixed samples of zinc cement between a glass plate and a slab of dentin to keep the excess moisture inherent in the mixing within the mix. This procedure kept moisture in the atmosphere from the mix. The authors studied the mix after two days with a scanning electron microscope. They found no intimate bond with the tooth and that hopeite crystals had formed on the cement. The authors also found that the body of the cement was completely porous. The pores present in the cement ranged in size from 1000A to 4000A.

Brannstrom and Nyborg (1971) studied silicate cement and composite resin. They were trying to determine whether a sealant prevented marginal leakage around the silicate cement and composite resin. The authors stated that marginal leakage allowed bacterial infiltration around and beneath the filling materials. The acid content of the silicate cement and the leakage around the composite resin were thought to cause the pulpal

damage in teeth with these types of restorations. The authors used 106 pairs of contralateral premolars slated for extraction for orthodontic considerations. Preparations were made in both teeth. One preparation was coated with a cavity liner (Tubulitec) and the other was not coated. Silicate cements were placed in 40 pairs of teeth. Forty teeth had the liner and 40 did not have the liner placed. Sixty-six pairs of teeth were used to test the composite resin. The pairs of teeth were extracted at various times from 1 to 8 weeks. The teeth were sectioned, and the sections were stained with Gram stain. Some sections were stained with Haematoxylin and eosin stain for the pulp. They found bacteria under almost all the unlined fillings of both types. The authors also determined that there was only pulpal inflammation when there was bacteria present under the fillings. Also, there was no inflammation when there were no bacteria present under the lined or unlined cavities.

Brannstrom and Nyborg (1974) studied bacterial growth and pulpal changes under lead inlays cemented with zinc phosphate cement and Epoxylite CBA 9080. The sample consisted of 29 pairs of young premolars to be extracted for orthodontic reasons. These teeth had preparations made in them. The preparations had a liner (Tubulicid) placed. One of the pair of cavities had the lead inlay cemented with zinc phosphate cement. The teeth were extracted after 3 to 4 weeks. No bacteria were observed beneath the inlays cemented with zinc phosphate cement. There was no pulpal inflammation beneath the zinc phosphate cemented inlays. One tooth cemented with zinc phosphate cement over an exposure showed no inflammation. Thirteen of the 20 cavities cemented with Epoxylite demonstrated bacteria beneath them. Twelve of these teeth exhibited

inflammation. The authors found no inflammation in the pulps under zinc phosphate cement if there were no bacteria present. They state that zinc phosphate cement per se does not irritate the pulp. The authors attributed the pulpal inflammation seen in earlier studies by other authors to poor debridement of the cavities or to marginal leakage that allowed bacterial infiltration under the fillings, and not to the reason stated by those other authors, which was chemical assault by the zinc phosphate cement on the pulp.



## CHAPTER III

### METHODS AND MATERIALS

#### A. Selection and Preparation of Specimens

Premolars extracted during orthodontic treatment were collected from the Oral Surgery Department, Loyola University School of Dentistry, Maywood, Illinois. The freshly extracted teeth were placed in sterile normal saline and refrigerated until they were prepared as specimens. Only teeth without caries and without restorations were used.

In this study, a total of twenty teeth were used. Each sample (Control, ZOE,  $ZnPO_4$ , and  $Ca(OH)_2$ ) consisted of five teeth. Each tooth was sectioned into halves. The mesial and distal of each section were treated as separate specimens. Therefore, each tooth yielded four specimens. The five teeth in each sample yielded a total of twenty specimens per sample.

The teeth were sectioned transversely approximately two millimeters from the cemento-enamel junction through the root with a high speed air rotor handpiece with water spray. The specimens were mounted to facilitate sample handling during preparation and testing. The specimens were embedded in COE (COE Laboratories, Chicago, Illinois 60658) tray plastic (powder/liquid ratio=8.0 g./4.0 ml.) utilizing a Buehler (Buehler Ltd., 2120 Greenwood Street, Evanston, Illinois 60204) specimen mounting press #9-22-67-166 with Dentsply Silicone Spray (Dentsply International York, Pa.) to facilitate the removal of the specimens from the mold.

The mounted specimens were ground with Buehler pressure sensitive Carbimet Paper Strips (#30-5160AB) grit numbers 240, 320, 400, and 600 stepping from coarsest to finest on a Buehler Handimet Grinder (#39-1470AB) with a continuous water flow over the grinding surface. The mounted specimens were rotated 90 degrees between grits and ground at a right angle to the last grind marks on the specimen. This grinding procedure kept the testing surface and the base surface of the specimen parallel. Final polishing was on a Buehler Handimet Polishing Table (rotating table) covered with AB Microcloth (#40-7208) and wet with AB Miromet polishing compound (#40-6355). The resulting specimens had a smooth, even, grassy appearance and exhibited an absence of scratches at 673 magnification.

The five teeth used in each sample were sectioned and mounted in plastic in succession during one day. The ten mounted sections were then stored in a room temperature humidior. All ten sections comprising the sample were then ground, polished, and numbered in succession on another day. They were again stored in a room temperature humidior. During one day, ten pre-treatment measurements per specimen were performed in succession on the ten sections (two specimens per section). Immediately after the last pre-treatment measurements were recorded for the sample, the dental base was applied to all the specimens in the sample. The specimens were then stored in a room temperature humidior for twenty-eight days. On the twenty-ninth day the dental bases were removed from the sections in succession. Immediately after the base had been removed from the section, ten post-treatment measurements per specimen were performed and recorded. All of the post-treatment measurements on the sample were completed that day.

B. Knoop Hardness Determination Method

A Kentron Micro Hardness Tester (model AK, #7122-1) manufactured by Riehle Testing Machines (East Moline, Illinois) was used to measure the hardness of the dentin. It was equipped with a Knoop diamond indenter and used with a load of 100 grams. The load was applied to the sample for fifteen seconds. The length of the indentation was measured with a filar measuring device in the ocular of the tester at 673 magnification. The filar measurement was converted to microns by using a conversion factor (0.2068) supplied by the manufacturer. The micron length was converted to a Knoop hardness value using the Knoop Hardness Number Table supplied by the manufacturer.

The root sections were oval with the buccal-lingual dimension the greater. The mesial and distal areas of the root section were treated as separate specimens.

Dentin has a heterogeneous nature. Its hardness varies slightly in the same specimen. The greatest variations occur near the pulp and the cementum. The measurements were made approximately equidistant from the pulp and the cementum in a curved line following the contour of the section. Ten measurements were made per specimen. The average of these ten measurements was used as the pre-treatment Knoop hardness value of the specimen. After twenty-eight days the samples were measured again. The ten final measurements per specimen were between and in line with the first measurements. The average of these ten measurements was used as the post-treatment Knoop hardness value of the specimen.

C. Procedures and Materials Utilized to Affect Dentin

The first sample was a Control. Nothing was placed on the dentin. The specimens were placed in a room

temperature humidior with approximately 100 percent humidity for twenty-eight days. The specimens were wiped with wet cotton balls and then dried. The final Knoop values of the specimens were determined.

The specimens in the Zinc Oxide-Eugenol sample were treated with Caulk ZOE-B&T (L. D. Caulk Co., Milford, Delaware). Caulk ZOE B&T is a zinc oxide and eugenol dental base. The ZOE was mixed with the proportions of one milliliter of liquid per one gram of powder. The ZOE was mixed on a sterile glass slab with a sterile spatula for one minute. The ZOE was placed on the specimens so that the dentin was completely covered to a thickness of approximately one millimeter. The specimens were placed in a room temperature humidior with approximately 100 percent humidity for twenty-eight days. The ZOE was removed by flicking the junction between the ZOE and the mounting acrylic with a knife. The specimens were wiped with wet cotton balls, dried, and the final Knoop hardness values determined.

The specimens in the Zinc Cement sample were treated with Stratford-Cookson Company Zinc Cement, Zinc Oxyphosphate Type (Stratford-Cookson Co., Yeaton, Pennsylvania). Zinc cement is chiefly composed of zinc oxide and phosphoric acid. The cement was mixed with the proportions of one capsule of powder (packaged by Stratford Cookson Company) per eight drops of liquid from the liquid measuring bottle supplied by Stratford-Cookson Co. The cement was mixed on a cool (sixty-five degrees Fahrenheit), sterile glass slab with a sterile spatula. Eight drops of the liquid were placed on the slab. The capsule of powder was opened onto the glass slab and divided into sixteenths. The cement was mixed according to the manufacturer's directions. The resulting mix had a consistency suitable

for cementation of inlays or crowns. The specimens were placed in a room temperature humidior with approximately 100 percent humidity for twenty-eight days. The cement was removed by flicking the junction between the cement and the mounting acrylic with a knife. The specimens were wiped with wet cotton balls, dried, and the final Knoop hardness values determined.

The specimens in the Calcium Hydroxide sample were treated with Caulk Dycal (L. D. Caulk Co., Milford, Delaware). Caulk Dycal, a calcium hydroxide dental base, came packaged in base and catalyst tubes. Equal amounts of base and catalyst were mixed on a sterile glass slab with a sterile spatula for ten seconds. The Dycal was placed on the specimens so that the dentin was completely covered to a thickness of approximately one millimeter. The specimens were placed in a room temperature humidior with approximately 100 percent humidity for twenty-eight days. The Dycal was removed by flicking the junction between the Dycal and the mounting acrylic with a knife. The specimens were wiped with wet cotton balls, dried, and the final Knoop hardness values determined.

## CHAPTER IV

### RESULTS

The pre-treatment hardness value of the Control sample was 57.25 KHN with a standard deviation of 5.18 KHN. The post-treatment hardness value of the Control sample was 54.31 KHN with a standard deviation of 4.69 KHN.

The pre-treatment hardness value of the ZOE sample was 55.63 KHN with a standard deviation of 3.03 KHN. The post-treatment value of the ZOE sample was 55.05 KHN with a standard deviation of 4.14 KHN.

The pre-treatment hardness value of the Zinc Cement sample was 54.78 KHN with a standard deviation of 3.67 KHN. The post-treatment hardness value of the Zinc Cement sample was 55.98 KHN with a standard deviation of 3.52 KHN.

The pre-treatment hardness value of the Dycal sample was 54.95 KHN with a standard deviation of 3.53 KHN. The post-treatment hardness value of the Dycal sample was 59.26 KHN with a standard deviation of 2.92 KHN.

The mean change in the hardness of each sample was as follows:

The Control sample was -2.94 KHN with a standard deviation of 2.13 KHN.

The ZOE sample was -0.58 KHN with a standard deviation of 3.05 KHN.

The Zinc Cement sample was +1.20 KHN with a standard deviation of 1.14 KHN.

The Dycal sample was +4.30 KHN with a standard deviation of 2.33 KHN.

The starting hardness value of each sample (Control, ZOE, Zinc Cement, Dycal) was statistically compared (paired sample t-test) to the final hardness value of the sample (See table I.).

The Control sample became significantly softer ( $P < .01$ ).

The ZOE sample did not change significantly.

The Zinc Cement sample became significantly harder ( $P < .01$ ).

The Dycal sample became significantly harder ( $P < .01$ ).

The mean change of each sample was statistically compared to the other samples through use of the two sample t-test (See table II).

The change in hardness of the Control sample was significantly different from the ZOE, Zinc Cement, and Dycal samples'. The Control sample softened during the experiment.

The change in hardness of the ZOE sample was significantly different from the Control, Zinc Cement, and Dycal samples.

The ZOE sample's hardness did not change significantly during the experiment.

The change in hardness (softening) of the Control sample was significantly ( $P < .01$ ) different from the ZOE sample's (no change in hardness).

The change in hardness of the Zinc Cement sample was significantly ( $.01 < P < .05$ ) different (harder) than the ZOE sample's (no change in hardness).

The change in hardness of the Dycal sample was significantly ( $P < .01$ ) different (harder) from the ZOE sample's (no change in hardness).

The Zinc Cement sample's change in hardness was significantly different from the Control, ZOE, and Dycal samples' change in hardness. The Zinc Cement sample's hardness became significantly harder during the experiment. The Zinc Cement sample's change in hardness was significantly harder than the Control sample's change in hardness ( $P < .01$ ). The Zinc Cement sample's change in hardness was significantly harder than the ZOE sample's change in hardness ( $.01 < P < .05$ ). The Zinc Cement sample's change in hardness did not get as hard as the Dycal sample's change in hardness ( $P < .01$ ).

The Dycal sample's change in hardness was significantly different from the Control, ZOE, and Zinc Cement samples' change in hardness. The Dycal sample's hardness became significantly harder during the experiment. The Dycal sample's change in hardness was significantly harder than the Control, ZOE, and Zinc Cement samples' change in hardness ( $P < .01$ ).



TABLE I

STATISTICAL ANALYSIS UTILIZING THE PAIRED SAMPLE t-TEST  
EVALUATION OF THE CHANGE OF HARDNESS OF AFFECTED DENTIN

	Control	ZOE	Zinc Cement	Dycal
Pre-treat.				
Mean	57.25 KHN	55.63 KHN	54.78 KHN	54.95 KHN
Std. Dev.	5.18 KHN	3.03 KHN	3.67 KHN	3.53 KHN
	n=20	n=20	n=20	n=20
Post-treat.				
Mean	54.31 KHN	55.05 KHN	55.98 KHN	59.26 KHN
Std. Dev.	4.96 KHN	4.14 KHN	3.52 KHN	2.92 KHN
	n=20	n=20	n=20	n=20
Amount of Change	-2.94 KHN	-0.58 KHN	+1.20 KHN	+4.30 KHN
Std. Dev.	2.13 KHN	3.05 KHN	1.14 KHN	2.33 KHN
	n=20	n=20	n=20	n=20
"P"	P<.01	-----	P<.01	P<.01
"T"	-6.16	-0.86	+4.73	+8.25

TABLE II

STATISTICAL ANALYSIS UTILIZING THE TWO SAMPLE t-TEST  
 EVALUATION OF THE CHANGE OF HARDNESS OF AFFECTED DENTIN

	Control/ ZOE	Control/ ZnPO <sub>4</sub>	Control/ Dycal	ZOE/ ZnPO <sub>4</sub>	ZOE/ Dycal	Dycal/ ZnPO <sub>4</sub>
"P"	P<.01	P<.01	P<.01	.01<P<.05	P<.01	P<.01
"T"	-2.88	-7.68	-10.26	-2.48	-5.75	+5.34

## CHAPTER V

### DISCUSSION

The results of this study showed that untreated dentin softened in a room temperature humidior. This softening is not unexpected, since one could expect the organic component of the dentin to decompose in this environment. The dentin is composed of an organic (connective tissue) and an inorganic component. The inorganic component would not be affected by the storage environment, but the connective tissue would decompose at room temperature. The decomposition of the dentin would cause a deterioration in the properties of the dentin, including the properties affecting its hardness.

The ZOE treated dentin showed no change in hardness. It has been reported in the literature (Ripa, 1972) that the in vivo effect of ZOE is to increase the hardness of dentin. The reported in vivo hardening effect of the ZOE may have been caused by the pulpal response to the ZOE and not by a direct chemical effect on the dentin. The results of this in vitro experiment indicate that the direct effect of ZOE is not to cause a hardening of the dentin, but to prevent the dentin from softening.

The Control and the ZOE samples responded differently to the storage environment. The Control sample became softer, while the ZOE sample stayed the same. Whether the effect of the ZOE in preventing softening of the dentin was physical and/or chemical was not determined. The physical prevention of softening would be due to the ZOE sealing the dentin away from the environment

of the humidior. The air and humidity in the humidior could not reach the dentin due to the physical barrier of the ZOE. The chemical prevention of softening would be due to the ZOE reacting with the dentin and forming a compound or compounds that resist decomposition. The ZOE may fix the collagen in the dentin and prevent it from decomposing. The ZOE's effect may be a combination of a physical barrier to the humidior environment and a chemical fixing of the organic components of the dentin to render the dentin immune to softening.

Biven (1971) found that eugenol and eugenol containing root canal sealers hardened dentin in vitro. The storage environment used in his experiment was 37°C and 90% relative humidity, and the load used to make the Knoop measurements was 10 grams. The storage environment Biven used in his experiment could have dried the specimens, which would have increased their hardness. Totah (1942) stated that drying increases the hardness of dentin. The light load (10g.) used by Biven to measure the hardness increases the error and variation in Knoop hardness determination.

In this experiment, a 100 gram load was used to determine the hardness of the samples. To keep the amount of error in the measurements to a minimum, a maximum load was used. When the indentation was measured with the filar ocular, the end of the indentation could be anywhere within the thickness of the filar lines. This variation in measurement of a long indentation had proportionally less error than on a short indentation. The amount of error in measurement in long and short indentations would be the same (the thickness of the line). The error is proportionally less in a long indentation than in a short indentation, because, as an illustration, two filar units

variation in an indentation 800 filar units long is proportionally one-half of two filar units variation in an indentation 400 filar units long. Therefore, the maximum convenient load was used. The 100 gram load placed most measurements at 700-800 filar units long, whereas Biven's 10 gram load placed the measurements at 200-300 filar units. Hegdahl and Hagebo (1972) found that hardness is load dependent and that increasing the load reduced the variation in measurements.

Zinc phosphate cement treated dentin showed an increase in hardness. The zinc phosphate cement not only prevented the dentin from softening, it also hardened the dentin. This result contradicts the supposition that the acid present in the cement is deleterious to the dentin (Van der Lehr, 1970). The hardness of the dentin increased due to the zinc phosphate cement treatment. This finding, when considered with the finding by Brannstrom and Nyborg (1971&1974) that zinc phosphate cement is not irritating to the pulp, indicates that zinc phosphate cement is still useful as a dental base.

The  $\text{Ca(OH)}_2$  treated dentin showed an increase in hardness and also caused the greatest increase in hardening of the dentin. The dentin treated with the  $\text{Ca(OH)}_2$  hardened three times as much as the dentin treated with the zinc phosphate cement. Ripa (1972) found that  $\text{Ca(OH)}_2$  increased the microradiodensity of dentin in vitro. He attributed the increase to calcium precipitation into the dentin from the  $\text{Ca(OH)}_2$ , which could be a possible explanation for the increased hardness. Mjor (1962) found that  $\text{Ca(OH)}_2$ , in vivo, increased the hardness of dentin. Mjor (1967), found an increase in the microradiodensity of  $\text{Ca(OH)}_2$  treated dentin in vivo. Based

on these investigators's research and the investigation described in this paper, one could state that  $\text{Ca(OH)}_2$  dental bases would be better because they show a greater increase in the hardness of the dentin.

A review of the results are that the Control sample decreased in hardness, while the hardness of the ZOE sample did not change. The zinc phosphate cement sample increased in hardness, while the  $\text{Ca(OH)}_2$  sample showed the greatest increase in hardness.

## CHAPTER IV

### SUMMARY AND CONCLUSIONS

#### Summary

Extracted human premolars were sectioned, embedded in acrylic, polished, and microhardness tested. Three test materials, ZOE, zinc phosphate cement, and  $\text{Ca}(\text{OH})_2$  were tested. A control sample was also prepared and stored in a humidior under the same conditions as the treated samples. The pre-treatment and post-treatment measurements of each sample were compared using the paired sample t-test. The mean change of each sample was compared to the mean change of the other samples using the two sample t-test. The control sample became softer. The ZOE sample stayed the same hardness. The zinc phosphate cement and  $\text{Ca}(\text{OH})_2$  samples became harder. The  $\text{Ca}(\text{OH})_2$  sample increased in hardness the most.

#### Conclusions

1. The in vitro effect of ZOE is different from its reported in vivo effect. In vivo, the ZOE has been reported to increase the hardness of dentin. In vitro, the ZOE does not increase the hardness of the dentin, but the ZOE prevented the dentin from softening in the storage environment.
2. The in vitro effect of zinc phosphate cement is to increase the hardness of the dentin.
3. The in vitro effect of  $\text{Ca}(\text{OH})_2$  is to increase the hardness of the dentin. The  $\text{Ca}(\text{OH})_2$  increased the hardness of the dentin more than the zinc phosphate cement did.

APPENDIX

Control Sample

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 1A		Specimen 1B	
56.41	47.49	58.37	50.59
58.06	53.86	58.52	49.48
58.84	54.13	61.08	50.09
61.42	55.97	59.78	51.99
62.09	56.70	59.46	50.97
61.58	58.84	55.39	54.82
60.42	54.41	55.39	53.04
58.68	53.72	54.41	55.11
55.83	52.77	56.11	54.55
57.46	49.48	55.39	55.11
Specimen 2A		Specimen 2B	
52.51	54.82	50.84	53.99
52.64	55.25	50.46	56.11
54.55	55.25	49.73	56.11
54.69	51.99	51.60	55.83
55.83	51.09	53.72	53.31
53.04	50.46	52.91	53.44
51.99	51.86	58.37	50.09
56.70	47.16	58.37	49.48
58.06	52.12	59.46	44.68
59.78	50.33	59.62	44.26



<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 3A		Specimen 3B	
51.09	52.24	53.31	51.35
50.46	52.64	56.70	53.72
51.47	52.64	56.70	52.24
49.24	49.48	55.83	53.04
51.35	44.17	55.83	52.12
53.58	48.18	56.70	51.99
55.68	43.86	55.55	51.09
54.27	49.12	54.27	52.64
56.86	41.08	53.58	52.77
56.41	44.99	54.69	51.09
Specimen 4A		Specimen 4B	
49.97	47.84	52.77	53.31
51.35	46.88	52.64	54.41
53.17	50.59	53.31	49.00
51.99	49.73	56.41	49.36
51.99	48.18	41.08	51.99
51.99	49.36	36.61	51.99
54.55	45.73	53.58	53.04
51.99	49.36	39.91	52.91
48.07	47.04	53.31	51.99
49.73	55.31	53.31	53.04

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 5A		Specimen 5B	
47.26	50.33	52.91	49.12
50.21	51.99	52.64	50.71
49.36	49.00	53.72	53.86
49.97	51.99	52.91	53.99
48.07	49.60	51.99	51.99
42.39	48.30	55.54	53.04
43.07	45.94	53.44	48.88
50.97	47.38	54.69	55.54
51.99	51.99	54.69	49.24
53.58	47.26	54.69	51.22
Specimen 6A		Specimen 6B	
55.39	51.99	51.99	51.99
54.97	46.81	50.84	49.60
54.13	54.13	50.71	55.11
58.84	53.44	52.51	53.17
57.15	51.86	53.99	51.60
57.92	55.68	57.46	48.88
60.76	54.41	57.46	47.72
60.10	55.54	55.83	48.65
59.30	54.97	57.92	46.66
59.15	55.11	58.52	47.84

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 7A		Specimen 7B	
63.48	60.76	64.72	51.99
61.75	59.62	67.91	63.65
64.18	60.27	68.69	67.13
62.09	60.76	69.28	64.36
65.26	57.00	67.33	57.00
65.26	59.94	62.26	59.62
60.76	58.84	63.30	61.92
61.92	54.97	62.26	60.27
65.08	58.52	64.72	62.09
60.92	62.95	65.64	59.62
Specimen 8A		Specimen 8B	
65.64	62.43	62.78	67.13
69.47	65.26	62.95	67.91
64.18	58.06	65.64	67.91
63.83	57.15	71.53	66.00
57.31	60.76	70.71	65.08
61.25	59.15	69.68	70.50
64.54	59.46	67.91	59.94
66.00	60.27	66.17	64.72
67.91	61.08	64.54	61.08
62.26	59.30	67.91	61.58

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 9A		Specimen 9B	
55.68	53.04	58.99	57.76
56.41	53.31	60.42	54.82
59.78	46.88	44.99	61.75
59.46	49.97	51.99	61.53
64.18	49.97	55.97	59.46
53.58	47.38	55.11	48.53
48.41	49.00	56.26	51.99
46.53	57.31	63.83	51.99
58.37	58.37	57.46	56.26
61.08	47.16	58.37	59.94
Specimen 10A		Specimen 10B	
66.75	58.37	62.43	62.26
55.25	56.86	58.99	56.55
62.78	52.64	61.25	59.94
58.37	56.11	60.92	62.09
60.10	54.55	59.94	55.11
60.10	56.41	67.91	59.62
58.68	54.27	62.26	60.42
56.70	61.42	63.30	61.92
60.42	61.92	65.82	58.99
62.09	59.78	60.76	61.58

## ZOE Sample

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 1A		Specimen 1B	
51.99	41.44	51.99	49.84
52.51	47.16	53.72	52.24
54.13	49.97	55.54	55.83
55.25	51.99	57.61	57.61
54.69	51.99	57.31	53.04
57.46	53.04	57.76	66.38
56.55	56.26	55.68	52.77
57.76	54.69	55.25	55.54
57.76	54.41	51.99	51.99
50.46	54.13	51.09	54.13
Specimen 2A		Specimen 2B	
52.51	48.41	52.91	50.97
51.73	48.53	51.99	50.21
49.84	47.95	52.38	51.35
51.99	49.00	52.64	50.71
51.99	50.46	51.99	50.97
52.91	48.07	50.46	49.84
54.27	49.00	50.84	49.48
51.99	49.00	51.09	49.60
51.99	51.35	50.97	51.99
52.64	45.62	50.21	47.95

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 3A		Specimen 3B	
58.37	52.24	53.17	50.46
58.68	55.83	55.54	55.25
58.68	52.38	55.54	58.22
61.08	57.46	58.22	58.99
63.30	62.26	58.52	58.52
66.38	56.86	62.09	66.17
60.76	60.59	57.76	66.56
61.75	58.37	58.37	58.52
60.10	60.59	58.99	58.68
57.00	60.27	57.92	55.54
Specimen 4A		Specimen 4B	
55.25	54.55	54.69	51.99
55.39	55.97	55.25	51.99
57.46	55.39	56.11	53.17
55.54	55.68	56.41	57.00
55.68	55.97	60.27	59.62
57.46	57.76	61.58	59.78
56.55	56.11	65.64	59.78
56.26	54.97	56.86	58.99
53.99	52.64	56.86	53.17
53.04	51.22	57.92	53.31

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 5A		Specimen 5B	
54.55	52.12	49.73	52.38
54.82	51.99	55.68	52.77
52.64	49.48	52.12	67.91
51.99	50.09	54.13	53.99
53.04	47.04	53.99	53.86
51.99	49.60	54.97	54.97
49.73	47.49	53.86	51.99
52.64	47.61	53.58	51.99
53.58	47.84	52.77	49.60
51.99	48.18	55.68	52.91
Specimen 6A		Specimen 6B	
54.97	47.26	48.88	54.41
55.11	49.48	50.46	56.11
53.44	51.09	49.48	53.99
56.11	51.99	53.86	56.55
57.92	55.25	52.77	55.11
52.91	53.31	53.58	54.27
53.72	54.27	52.24	50.21
55.83	56.41	55.39	49.97
52.38	53.44	56.11	51.47
52.77	53.17	55.25	49.24

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 7A		Specimen 7B	
51.99	55.54	58.06	55.97
56.86	54.55	56.55	56.70
57.61	56.70	56.26	56.26
60.76	54.13	56.11	60.27
57.61	53.86	55.11	54.82
56.86	55.25	56.41	54.55
54.55	53.58	54.97	51.99
57.00	57.00	52.77	51.09
57.61	56.70	59.30	55.25
56.41	58.68	58.84	54.27
Specimen 8A		Specimen 8B	
53.99	48.76	52.64	51.99
54.41	51.99	53.72	55.11
56.26	48.65	50.09	53.58
54.13	53.04	53.99	54.55
55.25	55.97	53.58	51.99
54.97	56.86	61.58	50.46
55.83	53.99	58.68	56.41
56.26	55.54	57.31	51.99
55.54	55.97	54.41	51.99
54.97	54.27	54.27	53.72



<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 9A		Specimen 9B	
62.43	61.92	65.82	67.91
58.06	64.54	57.00	71.96
59.15	62.78	61.42	60.10
59.30	60.59	64.36	67.91
59.15	58.68	61.25	67.91
62.26	61.92	64.72	63.65
61.42	55.68	58.37	55.68
66.38	62.43	61.25	67.91
64.90	62.60	58.84	67.91
59.94	57.46	65.08	59.62
Specimen 10A		Specimen 10B	
53.17	52.12	49.24	49.84
51.99	51.99	56.11	51.99
46.24	52.77	54.82	51.99
50.84	54.97	55.54	53.44
45.62	54.13	57.15	54.82
51.60	53.58	55.25	53.58
54.27	50.33	56.86	62.60
56.26	53.31	55.11	57.76
54.41	79.73	56.55	53.44
53.31	114.09	55.68	51.99

## Zinc Phosphate Cement Sample

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 1A		Specimen 1B	
50.59	45.83	50.84	49.84
57.61	48.65	50.09	53.72
55.25	51.99	53.58	53.58
52.38	51.99	51.99	54.97
52.91	54.97	55.39	56.55
50.59	51.99	54.41	56.11
49.84	51.99	56.41	53.31
47.49	51.99	52.77	51.99
49.00	53.31	54.97	50.84
46.88	57.15	49.84	49.24
Specimen 2A		Specimen 2B	
59.30	60.92	56.41	57.92
55.68	59.78	60.27	57.46
57.15	57.46	51.99	55.39
58.06	58.68	56.41	56.11
54.55	60.42	51.99	54.97
55.97	53.86	55.54	56.70
55.83	56.41	52.91	60.76
55.97	55.83	58.52	56.70
46.88	58.06	56.41	59.46
57.46	60.42	64.00	57.31

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 3A		Specimen 3B	
48.41	58.06	58.52	62.26
49.24	52.77	57.15	58.37
50.09	55.39	53.86	61.75
56.26	55.11	57.46	53.86
53.04	52.38	52.64	60.27
49.97	55.97	56.41	56.26
50.71	55.25	60.10	56.55
51.99	51.99	55.54	55.11
54.55	49.60	53.72	54.55
53.31	51.99	50.84	54.27
Specimen 4A		Specimen 4B	
48.07	47.95	54.13	56.41
50.97	50.71	52.64	53.86
50.59	49.24	51.35	52.24
50.84	48.18	51.99	55.68
47.49	51.99	49.84	54.13
51.99	51.47	51.86	53.99
51.99	52.12	53.99	57.31
51.99	52.51	51.99	56.26
50.71	51.99	52.77	56.25
50.97	52.12	55.97	55.68

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 5A		Specimen 5B	
53.99	55.54	55.68	51.86
52.64	55.97	53.99	52.91
55.25	53.44	54.97	53.58
53.86	56.11	51.99	55.68
50.97	54.69	55.83	55.39
51.99	53.04	57.00	58.37
53.86	55.39	55.11	58.99
54.13	57.15	58.06	59.62
53.99	60.10	58.22	58.22
60.76	59.46	58.22	59.94
Specimen 6A		Specimen 6B	
51.99	54.27	56.11	55.54
53.86	52.24	52.77	57.92
55.83	56.86	56.11	58.68
53.99	52.24	57.61	62.09
52.51	55.11	54.55	62.09
55.39	55.39	56.11	60.92
54.27	54.41	59.62	59.94
54.97	56.86	58.84	56.55
53.44	58.06	56.86	59.94
52.64	54.82	55.68	59.94

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 7A		Specimen 7B	
50.21	48.30	51.99	54.97
50.71	48.88	48.76	49.00
45.09	50.21	49.00	54.27
47.72	54.27	53.72	55.11
51.09	49.60	51.99	53.31
51.47	48.76	54.27	53.58
50.46	47.84	47.49	47.72
52.12	47.84	45.94	48.07
51.99	52.77	50.59	55.83
44.47	54.97	50.71	53.31
Specimen 8A		Specimen 8B	
54.69	50.09	57.61	58.37
55.54	55.54	54.41	57.46
48.30	57.46	54.55	55.97
50.21	52.38	54.55	55.97
49.60	49.00	54.55	56.41
51.35	51.35	54.55	54.27
49.12	46.66	53.58	55.54
53.31	51.99	54.13	61.94
51.99	55.39	57.76	60.10
51.99	55.39	56.11	60.76

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 9A		Specimen 9B	
61.42	60.76	56.86	67.91
59.15	60.42	66.00	59.46
61.08	63.65	56.41	59.15
60.10	64.90	61.08	60.27
59.30	61.75	58.06	57.61
53.17	58.37	58.06	60.27
59.15	61.58	58.06	65.08
57.15	54.13	61.25	57.76
63.65	57.76	59.46	66.38
61.08	62.60	63.48	61.42
Specimen 10A		Specimen 10B	
54.13	62.78	67.91	61.08
55.39	62.09	66.56	65.08
53.58	60.76	63.48	62.60
58.22	60.59	62.43	64.72
58.84	57.92	62.43	63.48
60.10	57.92	62.60	62.60
59.15	55.39	65.64	63.65
61.08	52.64	63.48	63.65
60.76	50.84	62.26	60.10
60.27	49.97	62.26	61.08

## Dycal Sample

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 1A		Specimen 1B	
57.76	61.75	62.26	62.43
51.09	61.58	64.00	59.30
55.68	63.30	60.10	63.30
58.37	62.95	60.42	64.90
60.27	64.54	63.83	61.08
64.18	65.82	65.64	61.08
60.76	67.91	62.26	67.91
59.30	66.75	58.68	63.12
61.25	63.48	60.10	67.91
60.42	56.70	59.46	62.60
Specimen 2A		Specimen 2B	
51.99	61.58	57.31	64.72
55.97	61.42	62.09	63.12
54.41	61.25	54.55	63.48
58.37	65.64	59.46	62.43
60.42	66.17	59.15	61.92
60.10	63.48	55.97	68.49
55.68	60.27	61.08	64.72
54.97	63.30	59.62	64.00
54.82	63.65	55.54	62.26
55.11	62.60	57.46	66.17

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 3A		Specimen 3B	
59.30	54.97	55.68	60.10
56.26	54.82	52.91	55.39
57.61	59.15	53.58	61.08
51.99	53.99	56.26	56.26
54.27	55.39	52.64	57.92
50.59	51.99	51.99	59.78
49.12	56.41	54.69	55.97
51.47	56.26	59.78	61.58
52.64	57.46	52.77	53.86
52.64	57.61	51.99	57.76
Specimen 4A		Specimen 4B	
49.73	53.31	48.76	50.46
48.41	58.68	49.48	53.72
50.84	56.55	50.09	57.46
50.84	57.61	52.51	54.55
55.11	60.10	55.25	57.31
51.99	57.76	49.36	58.22
51.47	56.55	51.99	54.41
49.97	54.13	48.07	57.61
52.64	58.22	47.26	49.24
50.59	56.70	44.57	54.41



<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 5A		Specimen 5B	
47.61	59.62	56.26	57.92
49.60	58.99	59.30	57.31
49.36	60.59	45.41	58.06
52.91	58.52	53.86	56.11
49.84	58.37	53.99	57.61
51.99	59.46	55.83	59.15
55.68	56.11	55.39	60.10
53.72	59.30	51.99	61.42
55.11	52.51	54.82	63.30
45.51	58.99	53.58	52.77
Specimen 6A		Specimen 6B	
58.84	60.76	55.97	69.28
58.84	58.99	53.72	58.52
55.11	61.08	57.46	61.25
55.68	56.26	55.39	61.08
58.99	59.78	54.41	58.99
57.61	62.26	57.31	60.42
55.39	62.26	60.92	66.17
55.11	65.26	57.31	67.91
57.61	63.65	62.95	64.90
56.26	55.97	53.58	66.00

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 7A		Specimen 7B	
53.31	55.83	51.99	51.22
57.61	55.39	49.97	54.41
54.82	59.30	49.60	54.82
53.17	56.26	49.73	52.51
54.27	57.76	52.38	52.77
53.86	57.76	52.38	55.83
53.17	53.17	53.72	57.31
54.13	61.58	54.13	56.70
53.72	61.92	53.99	59.78
53.99	59.15	54.97	60.10
Specimen 8A		Specimen 8B	
53.58	63.12	56.86	57.00
56.86	59.94	58.99	56.26
55.97	59.94	54.69	60.92
48.76	60.76	56.86	58.06
52.38	59.78	56.86	61.08
55.11	55.68	53.86	58.99
51.09	56.26	52.91	60.27
54.69	56.55	57.00	56.26
57.15	58.68	53.99	52.38
56.26	56.41	58.52	63.12

<u>Pre-treat.</u>	<u>Post-treat.</u>	<u>Pre-treat.</u>	<u>Post-treat.</u>
Specimen 9A		Specimen 9B	
57.31	60.76	60.59	61.58
51.99	59.15	61.25	62.43
49.73	56.70	59.94	61.08
52.77	53.99	59.30	64.36
51.99	56.86	59.30	58.68
51.35	58.06	58.06	56.86
51.73	50.71	59.78	60.92
54.97	56.26	56.55	59.62
54.82	56.11	60.59	60.42
53.17	54.13	60.59	60.76
Specimen 10A		Specimen 10B	
49.73	53.86	55.97	60.76
50.84	62.60	59.46	58.22
53.86	62.60	59.15	55.39
53.44	58.37	59.30	55.54
55.25	60.27	59.46	58.37
52.77	57.46	57.92	63.30
57.76	61.75	56.55	60.42
56.41	56.26	54.82	63.48
55.83	53.17	56.11	59.15
56.26	51.99	54.41	62.78

## BIBLIOGRAPHY

- Batchelor, R. and Wilson, A. D. Zinc oxide-eugenol cements: I. The effect of atmospheric conditions on rheological properties. Arch. Oral Biol., 7:333-336, 1969.
- Biven, G. M., Bapna, M. S., and Heuer, M. A. The effect of eugenol and eugenol-containing root canal sealers on the microhardness of human dentin. Masters thesis, Loyola University, Chicago, Illinois, 1971.
- Brannstrom, M. and Nyborg, H. The presence of bacteria in cavities filled with silicate cement and composite resin materials. Swedish Dent. Jour., 64:149-155, 1971.
- Brannstrom, M. and Nyborg, H. Bacterial growth and pulpal changes under inlays cemented with zinc phosphate cement and Epoxylite CRA 9080. Jour. Prosthet. Dent., 31:556-565, 1974.
- Cartz, L., Servais, G., and Rossi, F. Surface structure of zinc phosphate dental cements. Jour. Dent. Research, 51:1668-1671, 1972.
- Council on Dental Materials and Devices. Council adopts American Dental Association specification No. 8 (dental zinc phosphate cement) and (agar impression). Jour. Amer. Dent. Assoc., 74:1565-1569, 1967.
- Craig, R. G. and Peyton, F. A. The microhardness of enamel and dentin. Jour. Dent. Research, 37:661-668, 1958.
- Craig, R. G., Gehring, P. E., and Peyton, F. A. Relation of structure to the microhardness of human dentin. Jour. Dent. Research, 38:624-630, 1959.
- Dennison, J. D. and Powers, J. M. A review of dental cements used for permanent retention of restorations Part 1: composition and manipulation. Jour. Mich. Dent. Assoc., 56:116-121, 1974.

- Ehrenreich, D. W. A comparison of the effects of zinc-oxide eugenol and calcium hydroxide on carious dentin in human primary molars. *Jour. Dent. Child.*, 35:451-456, 1968.
- Garberoglio, R. and Brannstrom, M. Scanning electron microscopic investigation of human dentinal tubules. *Arch. Oral Biol.*, 21:355-362, 1976.
- Hegdahl, T. and Hagebo, T. The load dependence in micro indentation hardness testing of enamel and dentin. *Scand. Dent. Jour.*, 8:449-452, 1972.
- Hodge, H. and McKay, H. The microhardness of teeth. *Jour. Amer. Dent. Assoc.*, 20:227-233, 1933.
- Hodge, H. Hardness tests on teeth. *Jour. Dent. Research*, 15:271-279, 1936.
- Mjor, I. A., Finn, S. B., and Quigley, M. B. The effect of calcium hydroxide and amalgam on carious vital dentine. *Arch. Oral Biol.*, 3:283-289, 1961.
- Mjor, I. A. Histologic studies of human coronal dentine following the insertion of various materials in experimentally prepared cavities. *Arch. Oral Biol.*, 12:441-452, 1967.
- Molnar, E. J. Residual eugenol from zinc oxide-eugenol compounds. *Jour. Dent. Research*, 46:645-649, 1967.
- Norman, R. D., Swartz, M. L., Phillips, R. W. and Raibley, J. W. Direct pH determination of setting cements. 2. The effects of prolonged storage time, powder/liquid ratio, temperature, and dentin. *Jour. Dent. Research*, 45:1214-1219, 1966.
- Outhwaite, W. C., Livingston, M. J., and Pashley, D. H. Effects of changes in surface area, thickness, temperature, and post-extraction time on human dentine permeability. *Arch. Oral Biol.*, 21: 599-603, 1976.
- Ripa, L. W., Guzman, C., and Dilzell, W. The effect of calcium hydroxide and zinc oxide-eugenol on dentine in extracted human teeth. *Oral Surg.*, 34:531-537, 1972.

- Rotberg, S. J. and deShazer, D. O. The complexing action of eugenol on sound dentin. Jour. Dent. Research, 45:307-310, 1966.
- Servais, G. E. and Cartz, L. Structure of zinc phosphate dental cement. Jour. Dent. Research, 50:613-620, 1971.
- Sturdevant, C. M., Barton, R. E., Brauer, J. C., and Harrison, M. L. The art and science of operative dentistry. McGraw-Hill Book Company, 1968, pp. 100-101.
- Swartz, M. L., Phillips, R. W., Norman, R. D., and Niblack, B. F. Role of cavity varnishes and bases in the penetration of cement constituents through tooth structure. Jour. Prosthet. Dent., 16:963-972, 1966.
- Totah, V. P. Increase in hardness of dentin on drying. Jour. Dent. Research, 21:99-101, 1942.
- van der Lahr, W. N. Let's toss out zinc phosphate cements. South Carolina Dent. Jour. 28:16-18, 1970.
- Wolf, O., Gedalia, I., Reissstein, I., Goldman, J., and Stieglitz, H. Effect of addition of  $\text{CaFPO}_3$  to a zinc oxide-eugenol base liner on the microhardness and fluoride content of dentin. Jour. Dent. Research, 52:467-475, 1973.

APPROVAL SHEET

The thesis submitted by Dr. Michael S. Borzello has been read and approved by members of the Department of Oral Biology.

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 with reference to content, form and mechanical accuracy.

The thesis is therefore accepted in partial fulfillment of the requirements for the Degree of Master of Science.

April 20, 1978

Date

A. Wayne E. Miles

Signature of Advisor