

Loyola University Chicago [Loyola eCommons](https://ecommons.luc.edu/)

[Master's Theses](https://ecommons.luc.edu/luc_theses) **Theses Theses** and Dissertations **Theses** and Dissertations

1979

Dimensional Accuracy of Epoxy Resins and Their Compatibility with Impression Materials

Daniel R. Aiach Loyola University Chicago

Follow this and additional works at: [https://ecommons.luc.edu/luc_theses](https://ecommons.luc.edu/luc_theses?utm_source=ecommons.luc.edu%2Fluc_theses%2F3006&utm_medium=PDF&utm_campaign=PDFCoverPages)

Part of the [Oral Biology and Oral Pathology Commons](http://network.bepress.com/hgg/discipline/652?utm_source=ecommons.luc.edu%2Fluc_theses%2F3006&utm_medium=PDF&utm_campaign=PDFCoverPages)

Recommended Citation

Aiach, Daniel R., "Dimensional Accuracy of Epoxy Resins and Their Compatibility with Impression Materials" (1979). Master's Theses. 3006. [https://ecommons.luc.edu/luc_theses/3006](https://ecommons.luc.edu/luc_theses/3006?utm_source=ecommons.luc.edu%2Fluc_theses%2F3006&utm_medium=PDF&utm_campaign=PDFCoverPages)

This Thesis is brought to you for free and open access by the Theses and Dissertations at Loyola eCommons. It has been accepted for inclusion in Master's Theses by an authorized administrator of Loyola eCommons. For more information, please contact [ecommons@luc.edu.](mailto:ecommons@luc.edu)
 @090

This work is licensed under a [Creative Commons Attribution-Noncommercial-No Derivative Works 3.0 License.](https://creativecommons.org/licenses/by-nc-nd/3.0/) Copyright © 1979 Daniel R. Aiach

DIMENSIONAL ACCURACY OF EPOXY RESINS AND THEIR

COMPATIBILITY WITH IMPRESSION MATERIALS

by

Daniel R. Aiach

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

November

DEDICATION

To my father and to my mother for offering their greatest love, support, and patience throughout all my life.

To my brothers and sister, for their encouragement help and understanding.

ACKNOWLEDGEMENTS

The author would like to express his appreciation and offer special recognition to Dr. James L. Sandrik, who as my advisor, offered invaluable inspiration during the course of this investigation.

I wish to thank Dr. William F. Malone, whose continual guidance throughout the period of my graduate education, removed for me the barrier between theory and clinical study and gave me the opportunity to spend the most valuable years of my education.

I also express my deep gratitude to Dr. Vikram Tripathi for his helpful assistance and friendship during the course of this investigation. ,

The author, Daniel R. Aiach was born, February 18, 1945, in Boghari, Algeria, to Emile and Florence Aiach.

In 1962, he graduated from the Lycee Michelte High School in Paris, France. He entered the Faculty of Medicine of Paris where he received a Certificate Preparatory to the Medical Studies.

He began his formal dental studies at the Faculty de Chirurgie Dentaire de Paris and received his degree from the Faculty of Medicine of Paris in 1969.

He entered the French Army and served as a dentist for a period of one year. He practiced general dentistry as a private practitioner in Paris for a period of seven years.

In 1977, he entered Loyola University School of Dentistry in a two year graduate program leading to a Certificate of Specialty in Prosthodontics and a Master of Sciences in Oral Biology.

VITA

iv

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

 $\bar{.}$

viii

Table

Ì

Page

CHAPTER I

INTRODUCTION

The accuracy of the indirect technique for the construction of crown and bridge restorations has been the subject of many studies. Restorations can be fabricated to accurately fit the prepared abutments by following a carefully prescribed technique. Many steps are involved in the procedure, each of which is a potential source of error. All die materials do exhibit some degree of dimensional change during setting. They also may exhibit low abrasion resistance causing margins to be carved away during the fabrication of the wax pattern. They may be incapable of reproducing the details in the wax pattern accurately. This could be due to the inherent surface roughness of the material or a lack of compatibility between die and impression material. Although laboratory tests for physical properties of materials do not always predict the clinical results that can be expected, they do give some indication of how well a certain material will behave in a specific situation.

ties: A successful dental die material should have the following proper-

- 1) High strength-hardness, and abrasion resistance in order not to damage the die during laboratory procedure.
- 2) Ability to reproduce details recorded by the impression material.
- 3) Ability to produce a die of accurate dimension; once the material is set it must be dimensionally stable over a period of time and unaffected by changes in temperature.

4) Compatibility with impression materials.

5) Easy to handle, and non-toxic.

6) Its cost should not be prohibitive.

Traditionally die stones based on autoclaved calcium sulfate hemihydrate have been widely used for the construction of dies. This is because of their acceptable dimensional stability. The reason for this approach is the predictable behavior of dental stones. Dental stones have low cost, compatibility with all types of impression materials, ease of use, low coefficient of thermal conductivity and long history as a satisfactory die material.

Stone dies are known to fracture because of their low tensile strength. This is especially true during removal from some of the current elastomeric impression materials with high elastic moduli. Another property of die stone is its low abrasion resistance increasing the likelihood of removing some of the stone surface during the carving of the wax pattern. This is a common source of error unless extreme care is used. Therefore stone dies should be manipulated as little as possible and ideally should be waxed only once. Through the years many types of materials have been investigated for their possible use as a die material. These have included, dental amalgam, acrylic resins, silicophosphate cements and polymer containing die materials. Research has shown none of those materials satisfied all the criteria previously mentioned. The use of self-curing acrylic polymer had been suggested, but the shrinkage on polymerisation makes these materials unsuitable for the construction of accurate dies. The epoxy resins have only recently been employed as a dental material. Although they appear to show a great deal of promise,

sparse knowledge is available concerning: chemistry, their compabibility with impression materials and their dimensional accuracy. The literature concerning those materials is sparse indeed.

The current investigation will determine the dimensional stability of four different brands of epoxy resins. Modern impression materials used in fixed prosthodontics like polysulfide, addition silicone, and polyether will be tested concerning their compatibility with the different die materials. The detail reproducibility will also be evaluated as well as the compressive strength of epoxy resins.

CHAPTER II

LITERATURE REVIEW

1. General Aspects of Epoxy Resins

Epoxy resins were not synthesized until 1936 by the Swiss chemist Pierre Castan while searching for a curable plastic material suitable for use in dentistry. Castan used epichlorohydrin as a starting material which on condensation with diphenylolpropane (bisphenol A) in alkaline environment formed diglycidyl ether of bisphenol A, which is the simplest form of epoxy resin.

$$
CH_2
$$

CH₂ CH CH_2 CH_2 CH_2 CH_1 HO CH_2 CH_2

$$
CH_2
$$
—CH—CH₂– O \leftarrow 0–O \leftarrow 0–CH₂-CH—CH₂ + 2H CL

diglycidyl ether of bisphenol A.

The epoxy resins are thermo setting resins which may cure at room temperature and possess the unique characteristics in terms of adhesion, chemical stability and strength. The epoxy resins are characterized by the reactive epoxy or oxirane group $\begin{array}{c} \n 0 \setminus \n 1 \setminus \n 0 \setminus \n 0 \setminus \n 1 \setminus \n 0 \setminus \n$ polymerisation points. In this group the ring is in somewhat unstable

conditions prone to open and to combine with compounds having an available hydrogen. Cross linkage is easily accomplished.

The typical epoxy molecule is represented by the diglycidyl ether of bisphenol A. Such epoxy resins are viscous at room temperature and may be cured by use of a reactive intermediate to join the resin chain. For restorative materials, the epoxy compound should set at a relatively fast rate, therefore there are some modifications in the backbone in the epoxy chain. (The hardener may also be different). Example: molecule of glycidyl methacrylate

may be reacted by - monoacrylate, diacrylate,

cyanoacrylate, polyurethane, aliphatic amine (Von Fraunhofer).

For the use as dental die the diglycidyl ether of bisphenol A is reacted by a hardener which is usually a tertiary armine and the reaction is very slow.

2. Dimensional Change

One pure epoxy resin according to Bowen (1956) underwent a volumetric shrinkage during curing "about 2 per cent before gelation and about 2 per cent after gelation ". This shrinkage could be reduced by addition of fillers such as glass, asbestos, silica, and alumina. Bowen estimated that depending upon the filler resin ratio used, the shrinkage during cure would be about 0.6% before gelation and less than 0.5% after gelation. The addition of a small amount of anhydrous calcium oxide incorporated in 2 samples appeared to produce expansion during curing.

Lee and Neville (1957) gave the curing shrinkage of an epoxy resin at 0.91 per cent (0.31 linearly). The inclusion of 20 per cent silica decreased the shrinkage to 0.77 per cent (0.26 per cent linear shrinkage). They attributed the decrease in curing shrinkage, when a filler was included, to the reduced peak exotherm temperature and to bulk replacement. Wasser (1962) used two filled epoxy resins Devcon W R And Devcon F 2 clinically. He only reported the linear shrinkage as given by the manufacturer to be 0.016 per cent and 0.05 per cent respectively. W. Kydd and Wykkuis in 1958 used epoxy resins as denture base materials, because epoxy resins have the property of remaining in a liquid state during molding. No fluid curing bath was required in processing the epoxy resin, and the procedure was carried out with dry heat at 120° F. The warpage index (difference in per cent change between molar to molar and flange to flange measurements) was only 0.05%, the heat cured prosthetic bases (methyl metacrylate) was observed to contract when cooled from polymerisation of about 0.99%.

Woelfel et al., in 1961, showed after 2 or 3 months in use or in water the epoxy resin bases were less stable in dimension and had a higher expansion when compared to the other techniques. The warpage index found was greatest for the epoxy resin bases and was found to be about 0.30 per cent.

Ostlund and Akesson, in 1960, used a detachable brass mold, painted with olive oil, and markers floating on the surface of the resin, to study the dimensional change of one pure epoxy resin (Epicote 815). They reported the shrinkage to be 0.027 per cent.

Cevitarese et al., in 1963, recorded the linear shrinkage of five denture base resins, including one epoxy. This data indicated shrinkage of the epoxy resin was much less than the shrinkage of the other resins when measured on denture like specimens.

Toreskog, Phillips and Schnell (1966) reported a greater curing shrinkage of two filled epoxy resins, Perma Rock (a silica filled epoxy resin) and Devcon F2 (an industrial modified epoxy). They stated the filled epoxy resins showed a greater shrinkage than what was reported. From a dimensional standpoint these materials would probably be clinically satisfactory if used soon after hardening. "If a die material required a long setting time it is possible that the dimensional change of the impression material might affect the accuracy of the resultant die. This should be borne in mind when die materials such as epoxy resins are being used."

Peyton (1965) in an evaluation of materials used for indirect

techniques stated the epoxy die material offer some difficulties in completely filling the cusp detail of the impression because of the tendency to entrap air as the resin mass is placed in the impression. He also declared epoxy resins require several hours to complete polymerisation at a controlled temperature and the polymerisation was inhibited by the presence of even a small amount of moisture. For this reason they cannot be used with hydrocolloid. He concluded that the properties of the resin materials have not been adequate to justify their extensive use and therefore improved dental stone and electro plated silver were the two most satisfactory die and cast materials.

Newman and William (1969) using epoxy resin as a die material, stated slight shrinkage occurred during polymerisation, resulting in tight-fitting inlays and crowns.

Gettelman et al., (1970) reported a polymerisation shrinkage of 0.03 to 0.14 per cent for filled epoxy resins and 0.10 to 0.20 per cent for methacrylate polymers. Roxby and Anderson (1972) testing five different materials showed two different polyester resins had a linear shrinkage after twenty-four hours of 0.27 per cent and 0.53 per cent.

Astiz and Lorencki (1969) tested eight different die materials used in order to control the expandion-contraction variable, an invar $metal¹$ was used for the construction of a master die and a master

 $¹$ a 36 per cent nickel iron product with an almost negligible coefficient</sup> of minimal expansion at room

casting. Reproduction dies were made from an impression taken of the master die. The master casting was oriented on the reproduction die and the space between the top of the slot in the master casting and the top of the reproduction die was measured. In testing an epoxy resin² die material they found the probability of contraction was greater than 0.60 per cent (p=.OS level).

Moser, Stone and Willoughby (1975) tested an epoxy resin (Epoxydent) and found the dimensional stability for this die resin compared favorably to another die stone (Velmix). In their study the use of dental stone as a control gave a contraction of 0.011 per cent. For the resin, the contraction varied from 0.064 to 0.079 per cent. They stated that since it is known epoxy resins shrink upon polymerisation whereas gypsum products expand upon setting their results probably tended to reflect small changes in the rubber impression material in addition to the dimensional changes of the die material itself.

Cavazos (1976) in a study of different die materials tested two epoxy resins: Pri Die and Dentsply. The per cent linear change was respectively 0.080 and 0.134 contraction. Simulated full crown dies from both brands of epoxy resin poured in polysulfide polymer, and polyether impressions, were slightly enlarged across the occlusal. He found the problem with the epoxy dies was the loss in radial dimension

2 Pri die (Jenlenko)

r

at the cervical and even more severe loss in height. The most accurate epoxy resin came from the polyether impression; there was no statistical difference between the dimensions of the epoxy dies from either of the two brands of resin. The occlusal dimension was nearly the same as that of the master die, the axial dimension at the cervical was almost 0.2 per cent less than that of the master die. Statistically, the impression material, polysulfide, or polyether, did not affect the radial dimension at either occlusal or cervical. There was a statistically significant difference between the height of the epoxy dies from these two impression materials, the master being the reference.

3. Detail Reproducibility and Compatibility with Impression Materials.

The ability of a die material to reproduce detail depends upon its inherent surface roughness as well as the compatibility of the die material with the impression material. If two impression materials have a similar detail duplication but a die material reproduces fine details better from the first impression rather than from the second, we can say that the die material is more compatible with the first impression.

Ostlund et al., (1960) suggested the use of an olive or silicone oil to isolate the rubber base impression, in order to prevent the sticking of the epoxy die. They mentioned epoxy resins could not be used with hydrocolloids impressions, since polymersation of the resin was disturbed by moisture.

Wasser (1962) used the epoxy resin Devcon with a rubber base impression. He also used a separating medium to prevent the impression to adhere to the die material. Toreskog (1966) found the compatibility

of filled epoxy resins and different impression materials tested was very poor. The use of polysulfide (Permlastic with Perma Rock) was the only combination with this die material, which yielded even a moderately satisfactory surface. Despite this fact he noted a decrease of 43 per cent from the control for the detail reproducability test. Permlastic was the only impression material that consistently gave a satisfactory surface with Devcon F2 die. The use of a silicone material (Polytrans with Devcon F2) gave a die with a surface slightly tacky but an average of six of the seven indentations could be seen after 24 hours for the detail reproducibility test.

Toreskog, Phillips and Schnell (1966) concluded the compatibility of the filled epoxy resin with the different impression materials tested was very poor. Gettelman (1970) stated the epoxies cannot be used with most impression materials, chiefly the silicones, nor with some rubber and all hydrocolloid and alginate materials as well. The explanation given was "the moisture retards the epoxy setting reaction and the agar materials are not stable for long enough to allow polymerisation to take place." Concerning the detail reproducibility he mentioned, the epoxy dies seemed to perform quite poorly; the polymer particles distorting the surface smoothness failed to reproduce fine details.

Moser, Stone and Willoughby (1975) found the reproducibility of detail of an epoxy resin (Epoxydent) to be excellent as measured by means of a test block, lines of 25 microns width were clearly reproduced. The die stone "Velmix" used for a similar test demonstrated poor

results and this was being probably attributed to the coarser particles size when compared to the resin material. They also found epoxy resin were compatible with most impression materials. In their clinical experiment they mentioned the use of a rubber base impression for the construction of a resin die material. No mention was made about the brand of the rubber base used, or the use or not of a separating medium applied to the impression prior to the pour of the die.

Cavazos concluded the epoxy resin could be used only with the polysulfide and polyether impressions. Neither of the epoxy resins tested was compatible with the silicone impression material (Elasticon), which prevented curing of the resin at the die impression interface, the color was also transfering from the impression to the die.

With one epoxy resin (Dentsply) bits of impression material were adhering to the .surface of the die. With the other (Pri Die) die surfaces developed tackiness. In the detail reproducibility test when an inert silicone mold was used, Dentsply epoxy reproduced the finest detail present a ridge of 1.5µm. in width Pri die reproduced a ridge of $3~\mu$. The other materials tested gave: silver $3.5~\mu$ m., stone $10~\mu$ m. When the epoxy resins were cured in molds made of the impression material, Pri die was equivalent to silver $(3.5~\mu$. to $10~\mu$.).

Loss of detail reproduction was observed when Dentsply was cured in polysulfide mold (Permalstic) 38. When Dentsply was cured in polyether and silicone (it was stated before by Cavazos that epoxy resin did not cure in silicone impression material) it had the same capacity of detail reproduction as the stone (Vel Mix) when used with any of the three impression materials $(6.19~\mu m.)$.

Vermilyea et al., (1979) using 3 different epoxy resins; Prie Die, Epoxydent, and Dentsply observed similar properties for the different resins. Early compressive strengths of the resins tended to be higher than those of improved dental stones. At 24 hours, however, the compressive strength of the resins and stones are comparable. Subjection of the die resins to pyrolysis revealed that the non-volatile inorganic filler content of Pri Die, Expoxydent, and Epoxy Die material were 49% 48%, and 26% by weight, respectively. The determination of dimensional accuracy using a brass model which represented a partially edentulous segment of dental arch showed that the linear shrinkage for Prie Die was 0.11 per cent at all times observed. For Epoxy die the contraction varied from 0.03 per cent at 2 hrs. to 0.10 per cent at 7 days. For Epoxydent expansion was observed initially then shrinkage occurred and ranged from 0.04 per cent at 24 hrs. to 0.11 per cent at 7 days. Further assessment of the dimensional accuracy was made using a plastic model of maxillary molar which was prepared to receive a full coverage restoration. Castings fabricated on 24 hrs. old Epoxydent dies were the only ones found to be acceptable when transferred to the prepared tooth. All dies aged beyond 24 hours were found to be unsuitable for precision fixed prosthodontics procedures. The detail reproducibility of all tested materials sucessfully conterreplicated a 0.025 mm line inscribed on a stainless steel block. The compatibility of the resins materials with the polyether (Polyjel) impression and silicone impression material (Xantopren) material was found to be acceptable, however,

resin materials cured against Citricon were soft and tacky. Polysulfide impression material (Permlastic) showed a tendency to adhere to the fine detail of the resin models. Resins poured against addition silicone impression material (President) showed macroscopic surface and subsurface porosity.

CHAPTER III

METHODS AND MATERIALS

From a standard stainless steel die (A.D.A. Specification 19) custom trays were constructed. Using these custom trays, three types of impression materials (Table I) were used: polyether, addition silicone, and polysulfide. Four different brands of epoxy resins were poured into those impressions and the specimens were measured after setting. All experiments and tests were conducted at room conditions.

•II I. I

(1) The stainless steel die -

This die with a highly polished surface (Fig. 1-2) had 2 vertical lines which were used to determine the accuracy of the impression and 3 horizontal lines which provided guidance and were used for the detail reproducibility evaluation. The distance between the vertical lines was found to be 2.4990 em.

(2) Custom trays -

From the metal die 12 acrylic* custom trays were used providing an optimum space of 3 mm. from the highly polished surface. The trays were provided with a lip (Figure 3) in order to help in positioning of the loaded tray on the die. The custom trays were constructed 15 days prior to their use. Six custom trays were used initially with the polysulfide rubber impression, while the remaining six were utilized with addition silicone impressions. After cleaning and sandblasting, the same trays

* Formatray - Kerr

were employed for the polyether impressions. A specific adhesive supplied by the manufacturer was used for each type of impression material, it was applied on the custom tray and allowed to set at least one hour prior to any impression taking procedure.

(3) Impressions -

The procedure of impression-taking of the die was identical for all the impression materials used. Freshly prepared impression materials were mixed according to the manufacturer's instructions. Prior to the mixture base and catalyst were weighed on a cent-o-gram triple beam (± 0.05g) balance model 311 (Ohaus Scale Corp.) using the following proportions:

1:0.14 polyether, base to catalyst ratio

1:1 addition silicone, base to catalyst ratio

1:0.5 polysulfide rubber, base to catalyst ratio

After the mixing was completed, the material was placed in a custom tray which was pressed on the die by means of a plastic plate. The plate, custom tray and die were maintained in position together using a C clamp (Figure 3). The temperature was recorded in the room with a glass thermometer and the relative humidity was recorded with a Micro-Hygrometer (The Microhygrometer by Air Guide). Finally the time was measured by a chronometer. After loading the custom tray it was introduced into a water bath maintained at 32°C. This bath was a full visibility jar bath blue M. (Blue M. Electric Company, Blue Island, Ill.) (Fig. 4) and was filled with dionized water. After the material had set, the readings

were recorded with the use of a Gaertner Travelling Microscope (Gaertner Scientific Corporation, Chicago, Ill.) (Fig. 5) graduated in 0.01 mm. increments with a magnification of x32. The impressions were then poured using the epoxy resins die materials which were allowed to set 18 hours at room temperature.

(4) Epoxy Resins -

The epoxy resins appear to be conventional systems employing a nonmetal filler, with an amine hardener. With epoxy resin (P) the manufacturer's instructions recommended the use of a silicone mold release with rubber impressions. (There were no further indications concerning the term rubber impressions). The epoxy base was stirred for 2 minutes, the base to hardener ratio was 10 to 1. After the addition of hardener to the base the mixture was mixed an additional two minutes. Epoxy R and C (Rock Model and Coe) were commercially packaged in a disposable container. The bases were supplied in small jars meant to be used with predosed hardener syringes. The bases were stirred 2 minutes before adding the hardener then the mixing was continued two more minutes before pouring the impression. For epoxy C the base was stored 5 minutes at 100°F. in a water bath before being stirred.

For epoxy D (Dentsply) the manufacturer's instructions stated the material was compatible with rubber base, polyether; compound and was impressions, but not with silicone or hydrocolloid. Even with these compatible materials the manufacturer recommended the use of a light spray of Dentsply die separator to prevent sticking of the die material to the impression. Epoxy D was mixed in a plastic cup for 3 minutes.

The base hardener ratio employed was 8:1. All die materials were poured using a vibrator for one minute, then hand centrifuged for one more minute. They were allowed to set 18 hours before the first reading was made.

The steel die was calibrated by making several measurements. The calibration was found to be 2.4990 em. Numerous trial specimens were made to familiarize the investigator with the manipulations and the reading technique.

The impression materials were then weighed and mixed according to the manufacturer's instructions. Precautions were taken to obtain an homogeneous mix and avoid bubbles. After the weighing and mixing the material was placed in the custom tray and positioned on the die. The custom tray was ,covered with a plastic plate and a"C" clamp held together the stainless steel die, the custom tray and the plastic plate. (Fig. 3) The next phase was to introduce the entire assembly into the water bath at 32° C. after two minutes and 30 seconds after the mix was started.(Fig.4) Polyether or addition silicone specimens were removed from the bath 6 minutes later. The time was increased beyond the present A.D.A. specifications. For the polysulfide rubber impression the specimen was removed after 10 minutes in the water bath.

Finally the impression was removed very carefully from the die, using a twisting and pulling motion. This precaution was taken to avoid discrepancies that could affect the accuracy of the impression. The impression was then placed on the Gaertner microscope. (Fig. 5) Five measurements were taken from which the mean was calculated. Repeated

lubrication of the microscope's drum with oil rendered more accurate readings.

Six specimens of each impression material were poured in each brand of epoxy die material. In order to avoid too much dimensional change when the polysulfide rubber impression material was used, three impressions were taken, measured and poured within one hour. The same procedure was then carried out in a similar manner with the three other impressions. A mold release was employed only for polysulfide rubber. For each impression material there was 24 impressions taken and poured in the different epoxy die materials. For each die material there were 18 samples originating from the three types of impression materials. (Fig. 6)

Compressive Strength -

Using bakelite test blocks (Fig. 7) six samples of each epoxy resin were obtained. Prior to pouring the die materials the bakelite blocks were sprayed with a silicone mold release. The samples obtained were cylindrical in shape, 3 mm. in diameter, 6 mm. in height. The Instron machine (Fig 8) was used to determine the compressive strength after 18 hours at a chart speed of 10 inch/minute and a cross head speed of 0.2 inch/minute. Epoxy C was tested at 24 hours, 48 hours, 72 hours, and 1 week using 6 samples at each time.

Top view of the stainless steel master die

stainless steel member if a

Drawing representing the characteristics of the stainless steel master die

I

The stainless steel die with the custom tray, and the plastic plate held together with the "C" clamp

FIGURE 3

Full visibility jar

p

FIGURE 4

FIGURE 5

Gaertner traveling microscope of impression

is)

FIGURE 6

Specimens obtained from the three type of impression materials

Bakelite block

FIGURE 8

Instron machine with specimen to be tested

CHAPTER IV

RESULTS

All impression materials were prepared and mixed at approximately the same room conditions. They were allowed to set in a water bath at 32°C. This was done to simulate as closely as possible open mouth temperatures. The epoxy die materials were then mixed and poured at approximately the same conditions of room temperature and humidity, in order to simulate the conditions of a dental office or commercial laboratory. The mean room conditions were 22° C. and the relative humidity was 58%.

The mean, standard deviation and percentage shrinkage of each die material at the different period is presented in Tables 2-6. This data is the individual average of 77 impressions taken from which 77 die specimens were obtained. Five recordings per impression were taken prior to the pouring of the die material, after which five readings per specimen were made at specific time periods.

After the experiment was completed, the first part, testing the polyether impression materials and epoxy (P) was repeated. (Table 5) This repetition was performed in order to determine that the technique was reproducible yielding no significant difference in result. It is important to notice that at the time the impressions were measured, the immediate accuracy of the different impression materials selected was similar. Table 6 shows the comparison between all die materials, illustrating their relative effect on the different impression materials.

The dimensional change (percentage contraction of each die material) as a function of time and as a relation to different impression materials have been plotted and are presented on figures 9-11. When the data was accumulated statistically using a "t" test (two tail probability at 0.05 level of significance) the significant differences between the die materials, when used with different impression materials can be seen. (Table 13-16)

Table 7 shows the compatibility between epoxy resins and the different impression materials. The detail reproducibility is reported in Table 8. The epoxy resin materials exhibit a similar resistance to compression, when tested for compressive strength at 18 hours. (Table 9) Epoxy resin C tested for compressive strength (Table 10) responded to change in time intervals of 24, 48, 72 hours, and 1 week, as shown in figure 12.

NAME, BATCH NUMBER AND MANUFACTURERS OF EACH MATERIAL

IMPRESSION MATERIALS

MANUFACTURER

Addition silicone:Reflect Kerr Company Romulus, Michigan

Polysulfide: Omniflex fast set Laboratories, Inc. Chicago, Ill.

EPOXY RESINS

(P) Pri Die: (R) Rock Model (C) Coe Die (D) Dentsply Die Jelenko Company Kuwata Pan Dent Corp. Livingston, New Jersey Coe Laboratories Inc. Chicago, Ill. Dentsply Company 62378 Experimental Experimantal 101077 100777

BATCH NUMBERS

PERCENTAGE CONTRACTION FROM IMPREGUM IMPRESSION

PRI DIE (P)

Impression Measurement Time Intervals

ROCK MODEL (RM)

TABLE 2 (Cont.)

PERCENTAGE CONTRACTION FROM IMPREGUM IMPRESSION

DENTSPLY (D)

Impression Measurement Time Intervals

PERCENTAGE CONTRACTION FROM REFLECT IMPRESSION

 \bar{z}

PRI DIE (P)

TABLE 3 (Cont.)

PERCENTAGE CONTRACTION FROM REFLECT IMPRESSION

DENTSPLY (D)

Impression Measurement Time Intervals

PERCENTAGE CONTRACTION FROM OMNIFLEX IMPRESSION

PRI DIE (P)

TABLE 4 (Cont.)

PERCENTAGE CONTRACTION FROM OMNIFLEX IMPRESSION

DENTSPLY (D)

Impression Time Intervals

PERCENTAGE CONTRACTION FROM IMPREGUM IMPRESSION

PRI DIE (P)

COMPARISON BETWEEN ALL DIE MATERIALS AND THEIR RELATIVE EFFECT ON THE DIFFERENT IMPRESSION MATERIALS.

IMPREGUM

COMPATIBILITY BETWEEN EPOXY RESINS AND IMPRESSION MATERIALS

Polyether and addition silicone are used without any mold release. Polysulfide are used with a mold release.

DETAIL REPRODUCIBILITY

Polyether and addition shown are used without mold release.

Polysulfide are used with mold release.

r

COMPRESSIVE STRENGTH MEASURED AFTER 18 HOURS FOR ALL EPOXY RESINS TESTED

I

COMPRESSIVE STRENGTH OF EPOXY (C) AT ALL TIMES OBSERVED

TABLE 11

OMNIFLEX		${\tt PD}$	$\mathbb{R}.$	\mathtt{C}	$\mathbb D$
PD	18 hrs.	$\pmb{0}$	$\boldsymbol{0}$	\mbox{O}	\mbox{O}
	48 hrs.	$\overline{0}$	$\mathbf 0$	$\mathbf 0$	$\mathbf 0$
	72 hrs.	0	0	$\mathbf 0$	$\boldsymbol{0}$
	1 wk.	$\boldsymbol{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$
RM	18 hrs.	$\mathbf 0$	$\boldsymbol{0}$	\mbox{O}	$\boldsymbol{0}$
	48 hrs.	$\boldsymbol{0}$	$\mathsf{O}\xspace$	$\boldsymbol{0}$	$\boldsymbol{0}$
	72 hrs.	$\boldsymbol{0}$	0	$\mathbf 0$	$\boldsymbol{0}$
	1 wk.	$\pmb{0}$	0	$\boldsymbol{0}$	$\boldsymbol{0}$
$\mathbf C$	18 hrs.	0	$\,0\,$	$\mathbf 0$	$\rm X$
	48 hrs.	$\mathbf 0$	\mathbf{X}	$\boldsymbol{0}$	$\mathbf X$
	72 hrs.	$\overline{0}$	$\mathbf X$	0	\overline{X}
	1 wk.	$\overline{0}$	\overline{X}	$\boldsymbol{0}$	\overline{X}
$\mathbb D$	18 hrs.	0	$\overline{0}$	$\boldsymbol{0}$	$\mathsf 0$
	48 hrs.	$\boldsymbol{0}$	0	$\mathbf 0$	$\mathbf 0$
	72 hrs.	X	0	$\boldsymbol{0}$	$\boldsymbol{0}$
	1 wk.	$\boldsymbol{0}$	0	$\mathbf 0$	$\boldsymbol{0}$

X - **Significant** at .05 level

0 - Not significant at .05 level

COMPARISON OF ALL DIE SPECIMENS ISSUED FROM THE POLYETHER AND ADDITION SILICONE IMPRESSION MATERIAL

There was no statistically significant difference observed among all the specimens at all times observed at P:.OS level.

(P) IMPREGUH

EPOXY RESIN (R)

IHPREGUM

No difference was recorded

REFLECT

EPOXY RESIN (C)

EPOXY RESIN (D)

FIGURE 9

TIME (hours

z \circ w a: ~ ~ u.. 0 < c :z (J

> a: w

FIGURE 10

0 w a: ::E ::E u. 0 c .
z (.)

a:

re de la companya de
La companya de la co

 \mathbf{l}

I

I

TIME(hours)

ra de la construcción de la cons

 \mathbf{I}

r

 $\overline{\mathcal{I}}$

T I ME (hours)

CHAPTER V

DISCUSSION

A. USE OF A TEFLON DIE

r

The first attempt to test epoxy resin materials was by the use of a teflon die which presented the same characteristics as the stainless steel die $(A.D.A.$ Specification $#19)$, which was later used. The epoxy resin material was poured directly in this teflon die. Although teflon material does not have the property to adhere to any material, it was preferred to use a silicone mold release in order to avoid damaging the die during the removal of the epoxy die specimens.

Use of the teflon die was not successful for the following reasons. First, the lines on the horizontal surface of the die were not accurate enough to give reliable measurements. For this reason it was very difficult to calibrate the die. Consistent readings were not able to be made and varied by as much as 0.0250 mm. Another factor which seemed to cause difficulty was the lamps around the microscope micrometer. The increase of temperature produced by the lamps modified somewhat the dimension of the die and produced an additional parameter in the study.

For these reasons the teflon die was discarded, however, it was felt that the fabrication of a teflon die with very thin, accurate and definite lines would be very helpful and could be the material of choice in testing eposy resins. The rise in temperature due to the lamps may

be controlled without excessive difficulty. The decision to use overnight curing time (18 hours) for this investigation was made for practical considerations to reflect its use in dental practice or commercial laboratory, although it is possible to obtain a satisfactory epoxy resin die in a time span of 2 or 3 hours. The time spans of 48, 72 hours, 1 week, correspond more likely to the time interval between pouring and use in the daily practice of dentistry.

Dimensional error of the impression materials was reduced by construction of acrylic custom trays prior to the investigation. Those custom trays were constructed at least 15 days prior to the experiment. Mold release agents were used only when the polysulfide rubber was employed. It was not necessary to use polyether and addition silicone with any mold release agent.

B. DIMENSIONAL CHANGE OF EPOXY RESINS

ra 1999.
David

This investigation was not directed toward testing properties of impression materials per se. Rather, materials were chosen which have been reported most acceptable in the literature for use in fixed prosthodontics. All impression materials presented a shrinkage. From this data, the polyether impression material seemed to be the most accurate followed by addition silicone, the polysulfide seemed slightly inferior. The "t" value comparing those impression materials did not disclose any statistically significant difference at $p=0.05$. This may be due to the high value of the standard deviation among all measurements of impression materials. All those materials may be considered as equally satisfactory after the initial set, as has been stated in the literature Craig, (1975)

Sawyer, (1974) and Ciesco, (1978).

re de la companya de
La companya de la co

All epoxy resins exhibited as expected some degree of shrinkage. The shrinkage observed varied depending upon the impression material used and the epoxy resin used. The least shrinkage was observed when epoxy resins were used with the polyether impression material, as a general rule. Epoxy C showed a mean linear shrinkage of 0.101% over one week period, followed by (D) epoxy 0.106 and (P) epoxy 0.115.

Epoxy (R) showed the most shrinkage with a mean value of 0.142 . Over the one week period epoxy C showed a slight expansion, which is observed for the most part between 18 hours and 48 hours. (This tendency to expand after 18 hours for epoxy C is found again when epoxy is used with the other impression materials). Epoxy D did not change and Epoxy (P) and (R) exhibited after 18 hours a slight additional shrinkage.

When addition silicone was used epoxy C showed a mean shrinkage of 0.127 per cent. Epoxy D, which exhibited the least shrinkage, after 18 hours (0.130 per cent) contracts then, and finally expands. When the non-lead polysulfide impression material was used, the shrinkage observed was much more significant with all epoxy resins tested. Epoxy R showed the least shrinkage 0.211 per cent followed very closely by C and D. Epoxy P showed a higher value.

The findings showed less shrinkage for epoxy, than was observed by other investigators (Bowen, 1956 Astiz and Lorencki, 1969). They are nearly similar to the results obtained by Lee and Neville, 1957 Kydd

and Toreskog, 1958 et al., 1966). In their investigation Ostlund et al., 1960 Willoughby 1975 showed a shrinkage which was much lower. An interesting fact is that in one of their methods, Willoughby et al., used a similar metal die from which a polysulfide rubber impression was taken and poured in Epoxydent resin. Their results appeared definitely different from the findings in this study, and the shrinkage of Epoxydent resin reported by means of two methods showed values of 0.064 to 0.079.

Cavazos, in testing the epoxy resins (P) and (D) found a mean value of 0.082 per cent shrinkage for (P) after 24 hours and 0.134 per cent shrinkage for (D) after 24 hours. This experiment gives somewhat more of an indication of applicable effect in clinical use. Cavazos who used a metal die from which he took an impression with an industrial silicone which was allowed to set for 2 months. We emphasized the use of a die material which is used to be poured into an impression material.

The statistical analysis did not show significant difference in dimensional accuracy between the different epoxy resin die materials at p=.OS level. Furthermore, there were no significant variations in the dimensional stability of any specimen from any brand when used with any specific impression material over one week period.

C. EPOXY RESINS AND IMPRESSIONS RELATIONSHIP

 \overline{r}

The question arises, why did more shrinkage occur when the epoxy die materials were used with polysulfide rubber? Is it due to the impression material itself or to the die material? There is no complete agreement in the literature upon the dimensional stability of polysulfide

over a 24 hour period. Several researchers (Craig, Sawyer et al, Ciesco) have stated polyether, addition silicone, and polysulfide rubber seemed to have the same comparative relative accuracy after 24 hours. Stone and Willoughby in using a method similar to ours in testing an epoxy resin (Epoxydent) concluded there were very small changes in the impression material. The polysulfide rubber exhibited a significant shrinkage over the setting period of the die material which is in agreement with other authors (Chong and Docking, 1969). Although the shrinkage of the impression material could be related to a reaction with the epoxy die materials, this was not likely as Stone and Willoughby reported acceptable dimensional stability of Epoxydent while using a rubber impression material. (No indication was provided concerning the brand of the impression material). Another hypothesis has been forwarded, the behavior of non-lead polysulfide may be different when in contact with epoxy resins. It was felt the long setting time of the die material fosters water loss from the non-lead polysulfide rubber. The opinion is substantiated by Phillips et al., and stated "If a die material required a long setting time it is possible that the dimensional change of the impression material might affect the accuracy of the resultant die. This should be borne in mind when die material such as epoxy resin are being used." Therefore the die material was poured in the Omniflex impression within 30 min. after the beginning of the experiment in order to avoid dimensional changes. There is a common agreement in the research concerning the dimensional stability of polyether and addition silicone over a 24hr. period. This seems in accordance with

the results where the dies specimens recovered from Impregum and Reflect rendered better values, than when they were compared to polysulfide.

red and the contract of the co

Die specimens poured in polyether impressions also showed less shrinkage than those poured in addition silicone impressions. It may be due to the hygrophilic activity of the polyether over the hygrophobic action of addition silicone. When the data was accumulated statistically using a "t" test two tail probability at 0.05 level of significance, one can see most of all epoxy resins used with polysulfide rubber lead to significant difference when compared with epoxy dies used with polyether or addition silicone Impregum or Reflect. (Table 11,12) The difference seemed to be reinforced as time passed.

D. COMPATIBILITY BETWEEN EPOXY RESINS AND IMPRESSIONS MATERIALS

The compatibility between epoxy resins and polyether and addition silicone appears to be excellent. (Table 7) No mold release was sprayed on the impression prior to pouring the die materials. After the epoxy resins had set and were separated from the impressions, these impressions appeared unaffected by the procedure and were ready to be poured a second time. The superior compatibility observed between addition silicone and epoxy resins differed from the results reported by Cavazos and Gettelman who had evaluated epoxy resins with condensation polymerisation silicones.

Using the same handling and centrifugation the dies obtained from addition silicone exhibited more superficial bubbles. However, the die specimens obtained from polyether and polysulfide were bubble free. This might be due to the contact angle of the impression material which is

very low (Lorren et al., 1976) and the non-wetting characteristics of the materials. In this matter, the addition silicone and condensation polymerisation silicone seem to behave similarly. This fact may also indicate very careful handling and centrifuging should be utilized when epoxy resins are poured in silicones. Bubble defects were not incorporated in the die materials poured in polysulfide material. But the use of a mold release was necessary and yielded a discolored die specimen. It seemed the colorant from the impression had impregnated the surface of the die material. Furthermore, the impression after being separated appeared aged and not ready for another pour.

Previous researchers like Toreskog et al., have found very poor compatibility between the epoxy filled resin and the different impression materials tested. In view of this experiment it seems that recent dental material technology has provided polyether and addition silicone material which present an excellent compatibility with all die materials, and most specifically epoxy resin.

E. DETAIL REPRODUCIBILITY

r

When polyether and addition silicone impression materials were used the detail reproduction recorded was excellent. (Table 8) In all cases the detail reproduction exceeded that required by the ADA specification Number 25 for gypsum products.

ADA specification No. 25 required reproduction of a groove in a test block rather than reproduction of a ridge on an elastomer surface, that ridge being the reproduction of a groove in a test block. All epoxy resins tested showed an excellent detail reproducibility. When

the polysulfide rubber impressions were used the utilization of a mold release altered the reproducibility at the surface of the die. This was observed mainly for epoxy (D) as it had been similarly reported by Cavazos. Epoxy (R) gave an excellent reproducibility as seen in Table 8. Epoxy (C) gave an acceptable reproducibility but the die specimens appeared stained. The color from the impression seemed transferring into the die material.

Those results are in accordance with the recent work of Willoughby et al., who found that the epoxy resins were reproducing detail better than the improved stone (Vel-Mix). In conclusion, it can be seen that the detail reproducibility is enhanced with polyether and addition silicone.

F. COMPRESSIVE STRENGTH

r

In table 9 it can be seen that epoxy (P) , (C) and (R) exhibited a similar compression resistance at 18 hours. Epoxy D showed a lowered compressive strength, however, the small sample size (3) may have been too small to draw any conclusions. Table 10 shows Epoxy C tested over a different period of time, statistically significant difference was found in the compressive resistance between the 18 hour specimens and the 1 week specimens when a "T" test was conducted at $p-.05$ level. This could mean that the material is not fully cured at 18 hours and becomes harder as time passes. It may explain the slight variation of the material observed in the data but the difference in compressive strength does not affect significantly the dimensional accuracy of the different

epoxy resins. As it has been shown in the statistical analysis, one significant property not studied was abrasion resistance. This important parameter ought to be evaluated to fully appreciate the utility of these die materials as substitutes for conventional gypsum products.

r

G. COMPARISON WITH OTHER DIE MATERIALS AND DISCUSSION CONCERNING A POSSIBLE USE OF EPOXY RESINS IN DENTISTRY

The die materials more commonly used in dentistry are the improved stones or type IV gypsum products. These materials exhibit a setting expansion which varied from 0.08% to 0.1% in good conditions of utilization. The improved stone materials compensate to a certain degree for the shrinkage exhibited by the impression materials, and consequently the stone die restore the approximate size of the master die. The silver plated dies when used exhibit a shrinkage which has been reported to vary from 0.7% to 1.3% (Vermilyea and Craig 1975) when polysulfide impression materials are used. The epoxy die materials exhibit a shrinkage during setting varying from 0.1% to 0.26% according to our data. As the polyether impression material contracts at approximately 0.16%, this will produce an additional shrinkage and the total contraction from the master die would be 0.42%.

The silverplated dies which exhibit greater contraction than epoxy resin die have been and are still widely used in fixed prosthodontics with very acceptable results, therefore the epoxy resin could be used with clinical success. Further research is necessary in order to
to determine the volumetric change of epoxy resin materials.

r an an A

The polyether impression materials seem to be the material of choice when epoxy resins are to be used. Addition silicone is accurate and equally satisfactory but due to its low contact angle, it presents more difficulties in handling. Therefore centrifuging is imperative. The use of colored die material may also facilitate the reading of the marginal limits.

CHAPTER VI

ra de la partida de la par

SUMMARY

A total of four epoxy resin die materials were tested for accuracy, dimensional stability, compatibility with different types of impression materials, reproducibility, and compressive strength. Using custom trays, impressions were taken from a stainless steel A.D.A. standard specification die for testing dental impression materials. After being taken, those impressions were measured and poured with epoxy resin die materials which were then evaluated at 18 hours, 48 hours, 72 hours and 1 week.

The impression materials were used in an environment that simulated the clinical conditions and were allowed to set in a 32°C water bath for 2 minutes longer than the manufacturer recommended. The epoxy die materials were used at a relatively constant room temperature and humidity. They were prepared according to the manufacturer's instructions. After setting 18 hours in room conditions the epoxy resin die materials were evaluated and subjected to statistical comparisons.

The conclusions of our investigation are as follows:

1. All epoxy resins tested were accurate and remained stable over the one week experiment when poured in polyether or addition silicone impression materials.

2. The detail reproducibility of epoxy resins and their compatibility with impression materials was excellent when polyether and addition silicone were used.

3. Differences among the various die materials were observed when the non-lead polysulfide impression materials were used. The use of a mold release agent which was found to be necessary with non-lead polysulfide impression material affected the detail reproducibility.

r an an Aonaichte a
D'an Aonaichte an A

4. The epoxy resins poured in the polysulfide impression materjal exhibited more shrinkage in comparison to their use with the other impression materials.

REFERENCES

r

- Astig, P.R. and Lorencki, S.F. Comparative Accuracy of Commonly Used Dental Die Materials, J. Canadian Dent. Assoc., 35:6pp. 320-324, 1969.
- Bowen, R.A. "Use of Epoxy Resins in Restorative Materials," J. Dent. Res. 35:360, 1966.
- Cavazos, F. Thesis, University of Indiana 1976 Comparative Study of Different Die Materials.
- Chevitarese, 0., Craig, R.G., and Peyton, F.A. Properties of Various Types of Denture Base Plastics, J. Pros. Dent., 12:711, 1963.
- Chong, M.P. and Docking, A.R. "Some Setting Characteristics of Elastomeric Impression Materials," Aust. Dent. J., 14:295-301, 1969.
- Ciesco, J. "A Comparison of the Accuracy and Dimensional Stability of Five Elastomeric Impression Materials" Thesis, Loyola University, April, 1978.
- Craig, R.G., O'Brien, W.T., and Powers, J.M. "Dental Materials, Properties and Manipulation," The C.V. Mosby Co., 1975.
- Gettelman, L. and Ryge, G. Accuracy of Stone, Metal, and Plastic Die Materials, J. of Calif. Dent. Assoc. 46:28, 1970.
- Kydd, W.L. and Wykhuis, W.A. A Modified Epoxy Resin as a Dental Base Material, J.A.D.A. 56:385-88, 1958.
- Lee, H. and Neville, K. Epoxy Resins in Their Application and Technology, New York, McGraw-Hill Book Company, Inc., 1957.
- Lorren, R.A., Salter, D.J., and Farihurst, C.H. The Contact Angles of Die Stone on Impression Materials, J.P.D., 36(2):176-180, August, 1976.
- Moser, J.B., Willoughby, G.m., and Stome, D.G. Properties and Characteristics of a Resin Die Material, J. Pros. Dentistry, 34:297, 1975.
- Newman, A. and Williams, J.D. Die Material For Inlay Crown and Bridge Work, Br. Dent. J., 187:415, 1969.
- Ostlund, S.G. and Akesson, N.A. Epoxy Resins as Die Materials, Odont. Revy 11:225, 1960.

r

- Peyton, F.A. Evaluation of Materials Used for Indirect Techniques, D.C.N.A., 213, March 1965.
- Phillips, R.W. Science of Dental Materials, 7th Edition, St. Louis, The C.V. Mosby Publishing Company, 1973.
- Toreskog, S. Properties of Die Materials, A Comparative Study, Thesis, Indiana University, 1964.
- Toreskog, S., Phillips, R.W. and Shell, R.J. Properties of Die Materials, A Comparative Study, J. of Pros. Dent. 16:119, 1966.
- Vermilyea, S.G., Huget, E.F., and Wiskoski II. J. Evaluation of Resin Die Materials, 42:, No. 3. p. 304 1979.
- Vermilyea, S.G., Powers, J.M. and Craig, R.G. Polyether, Polysulfide and·Silicone Rubber Impression Materials, Part 1, Quality of Silverplated Dies, J. Mich. Dent. Assn., 57:371, 1975 Part 2, Accuracy of Silverplated Dies, J. Mich. Dent. Assn., 57:405, 1975.
- Von Fraunofer; Scientific Aspects of Dental Materials, Butterworths, London and Boston, 1975.
- Wasser, V.E. New Die Material for Master Casts, J. Pros. Dent., 11:1122, 1961.
- Wasser, V.E. New Die Material for Master Models, D. Digest, 68:76, 1962.
- Woelfel, J.B., Paffenberger, J., and Sweeney, W.T., J.A.D.A. 62:643-57, 1961.

APPROVAL SHEET

The thesis submitted by Daniel R. Aiach has been read and approved by the following committee:

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

Dr. William F. Malone Professor, Fixed Prosthodontics, Loyola

Dr. Vikram D. Tripathi Research Staff Member of the U.S. Gypsum Company

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.

November 20 197 Date

f

1. Santut Directe