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## Effect of Time on Gypsum-Impression Material Compatibility

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LOMA LINDA UNIVERSITY  
School of Dentistry  
in conjunction with the  
Faculty of Graduate Studies

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Effect of Time on Gypsum-Impression Material Compatibility

by

John Boram Won

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A Dissertation submitted in partial satisfaction of  
the requirements for the degree  
Master of Science in Prosthodontics

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August 2012

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Each person whose signature appears below certifies that this thesis in his/her opinion is adequate, in scope and quality, as a dissertation for the degree Master of Science.

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## ACKNOWLEDGEMENTS

First, I would like to take this opportunity to thank God for giving me this opportunity to study, learn and share. I am living proof that through Him, all things are possible.

I would like to thank my committee members for their time, effort, invaluable advice and direction. Drs. Winer, Kattadiyil, Goodacre and Lozada, you have been a constant source of inspiration for me to strive to achieve in my professional and personal life. Each one of you has helped to educate me not just in the academic/research realm, but also in how to live my life as a Christian professional. I will always strive to teach my students as you have taught me.

I would like to especially thank Drs. Rishi Patel and Montry Suprono for your contributions to my research project. Your friendship and support are invaluable to me. It has been a pleasure working with both of you and I look forward to future projects together.

To Dr. Oyoyo and Laurita Shu, thank you both for your assistance in the statistical analysis. The completion of this thesis would not have been possible without your help.

Finally, to my family and friends, thank you for sharing my joys, my struggles, triumphs and failures. Thank you all for your support and encouragement. No accomplishment has worth unless it can be shared with those for whom you love and care.

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## ABBREVIATIONS

ANSI	American National Standards Institute
ADA	American Dental Association
No.	Number
$\beta$	<i>beta</i>
<i>d</i>	<i>delta</i>
CaSO <sub>4</sub>	Calcium Sulfate
H <sub>2</sub> O	Water
s	Sol
Ca <sup>2+</sup>	Calcium Ion
aq	Aqueous
SO <sub>4</sub> <sup>2-</sup>	Sulfate Ion
Na <sup>+</sup>	Sodium Ion
Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	Tetrasodium pyrophosphate
P <sub>2</sub> O <sub>7</sub> <sup>4-</sup>	Pyrophosphate Ion
$\alpha$	<i>alpha</i>
mm	Millimeter
$\mu\text{m}$	Micrometer
Hg	Mercury
SEM	Scanning Electron Microscope

## ABSTRACT OF THE DISSERTATION

Effect of Time on Gypsum-Impression Material Compatibility

by

John B. Won

Advanced Specialty Education Program in Prosthodontics  
Loma Linda University, School of Dentistry, August 2012  
Dr. Mathew T. Kattadiyil, Chairperson

The purpose of this study was to evaluate the compatibility of dental gypsum with three recently introduced irreversible hydrocolloid (alginate) alternatives. The test materials were Alginit® (Kerr™), Position Penta Quick® (3M ESPE™) and Silgimix® (Sultan Dental™). The irreversible hydrocolloid impression material, Jeltrate Plus antimicrobial® (Dentsply Caulk™) served as the control.

**Materials and Methods:** Testing of materials was conducted in accordance with ANSI/ADA Specification No. 18 for Alginate Impression Materials. **Statistical**

**Analysis:** The 3-Way ANOVA test was used to analyze measurements between different time points at a significance level of ( $p < 0.05$ )

**Outcome:** It was found that there was greater compatibility between gypsum and the alternative materials over time than the traditional irreversible hydrocolloid material that was tested. A statistically significant amount of surface change/incompatibility was found over time with the combination of the dental gypsum products and the control impression material (Jeltrate Plus antimicrobial®).

## CHAPTER ONE

### INTRODUCTION

Irreversible hydrocolloid (alginate) is the most commonly used impression material for creating dental casts in diagnosis, treatment planning, and fabrication of removable prostheses. Gypsum based products are the materials of choice of those in the dental field when fabricating casts of patients' oral structures. In recent years, several companies have introduced alginate alternative materials for making dental impressions. However, there is little published data on the gypsum compatibility of these irreversible hydrocolloid alternatives. Also, there is no published data on the quality of gypsum based dental casts that have been fabricated against irreversible hydrocolloid or its "alternatives" over a period of time. The testing was carried out according to Specification No. 18 of the American National Standards Institute/American Dental Association (ANSI/ADA) for Irreversible Hydrocolloid detail reproduction and gypsum compatibility.

The objectives of this in vitro investigation were to compare the compatibility of three different alginate alternatives for their gypsum compatibility using the parameters outlined in the ANSI/ADA Specification 18. An irreversible hydrocolloid (alginate) served as the control in this research. The gypsum casts were then graded at various time points for their detail reproduction stability over time.

The null hypothesis is that there is no significant difference in gypsum compatibility between irreversible hydrocolloid and the alternative impression materials and that there would be no change in the gypsum casts at different time points.

## CHAPTER TWO

### LITERATURE REVIEW

A colloid is described as “a solid, liquid, or gaseous substance made up of large molecules or masses of smaller molecules that remain in suspension in a surrounding continuous medium of different matter.”<sup>1</sup> Colloids have been described as the fourth state of matter. Somewhere between the definition of a solution and a suspension, we find the definition of a colloidal solution, otherwise referred to as a *sol*. A hydrocolloid is a colloid that contains water as the dispersion medium.

#### **Irreversible Hydrocolloid**

Irreversible Hydrocolloids (aka Alginates) are one of the most widely used impression materials in dentistry. It was first developed as a substitute for agar impression materials due to the scarcity of agar during World War II. Alginate is based on a natural substance that is extracted from brown seaweed called anhydro- $\beta$ -*D*-mannuronic acid or alginic acid. It remains popular due to its ease of manipulation, the comfort of the patient during impression making and because it is relatively inexpensive.

Diatomaceous earth particles increase the strength and stiffness of the alginate gel. They aid in forming the sol by dispersing the alginate particles in the water and help form a non-tacky, firm gel surface. Calcium sulfate dihydrate is used as the reactor and fluoride is added as an accelerator for the gypsum products that will be poured into the

impression. Sodium phosphate acts as a retarder to extend the working time of the impression material.

Many manufacturers also have added organic glycols in order to reduce the amount of silica dust particles, essentially making the powder “dustless.” This is due to the concern that inhalation of these particles over the long term may be a health hazard.

A soluble alginate serves as the chief active ingredient of irreversible hydrocolloid impression materials. It can be found in the form of potassium, sodium, or triethanolamine alginate. A sol is formed when the alginate is mixed with water. The viscosity of the sol is dependent upon the molecular weight of the alginate compounds, which varies upon their treatment method by the manufacturers. The composition of Irreversible hydrocolloid impression material powder is outlined in Table 1.

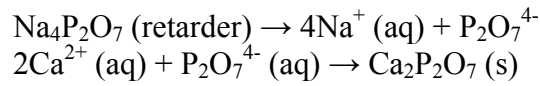
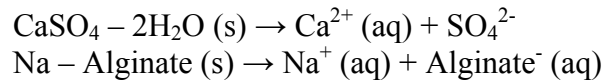
Buchan and Peggie<sup>2</sup> studied the effect of changing the concentration of the different ingredients in irreversible hydrocolloid impression materials. By altering the amount of the different components, they were able to analyze the effect on dimensional stability, hardness, elasticity, and setting time. Based on these principles, different manufacturers are able to change the composition of the alginate in order to achieve specific effects on the impressions that are made.

Table 1: Composition of Irreversible Hydrocolloids

<b>COMPONENTS</b>	<b>WEIGHT (%)</b>	<b>FUNCTION</b>
Diatomaceous earth or silicate powder	56	Controls the consistency of mixed alginate and flexibility of set impression
Potassium, sodium, or triethanolamine alginate	18	Dissolves in water and reacts with calcium ions.
Calcium sulfate dihydrate	14	Reacts with potassium alginate to form insoluble calcium alginate gel
Potassium sulfate, potassium zinc fluoride, silicate or borates	10	Counteracts the inhibiting effect of hydrocolloid on the setting of gypsum
Sodium phosphate	2	Reacts with calcium ions to extend working time before gelation
Organic glycols	Trace	Makes powder dustless
Wintergreen, peppermint, anise	Trace	Produces pleasant tastes
Pigment	Trace	Color
Disinfectants	1-2	Decreases viable organisms

The gelation process consists of the reaction of soluble alginate with calcium sulfate that leads to the formation of an insoluble calcium alginate gel. A polymer network is formed by calcium ions replacing the sodium or potassium ions on adjacent alginate molecules. This process is displayed in Figure 1. <sup>1</sup>





KEY	
s	sol
aq	aqueous

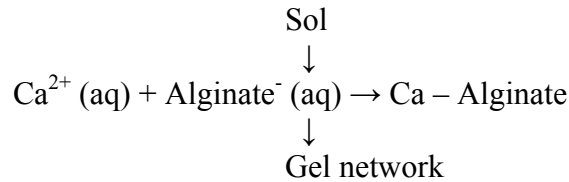


Figure 1: Displays the gelation process in irreversible hydrocolloid impression materials

When considering the working time of irreversible hydrocolloid impression materials, the presence of retarders are necessary because of the rapid production rate of calcium alginate. Therefore, a third water-soluble salt, in addition to calcium sulfate and soluble alginate, must be added (e.g. Trisodium phosphate). This creates a reaction between the calcium sulfate and soluble salt rather than the soluble alginate. Therefore, the production of calcium alginate is delayed until the trisodium phosphate is fully reacted.

In 1946, Skinner and Pomes<sup>3</sup> published on the dimensional stability of eight alginate impression materials that were available on the market at that time. It was found that seven out of the eight alginate impression materials had superior dimensional stability to those of reversible hydrocolloid when the materials were stored at 35-45% relative humidity. They also found that four of eight irreversible hydrocolloid impressions were dimensionally stable after being sealed in a bag at 100% humidity for up to 13 hours. They also described the creation of an insoluble gel over the alginate by

soaking the impressions in a metallic salt. It was also desirable to have an accelerator for the gypsum in this “fixing” solution to counteract the byproducts of syneresis that might affect the setting of dental stone. Over time, many manufacturers have incorporated the salts found in these “fixing” solutions into the powder of the alginate impression material.

In 1947, Skinner and Pomes<sup>4</sup> described the technique for manipulation and selection criteria for alginate impression materials. According to their research, the clinician’s ability to control the setting time of irreversible hydrocolloid impression material was limited to the temperature of the water used to mix the powder. They also found that the maximum strength of alginate material is not reached until two to three minutes after initial gelation had taken place.

An adequate thickness of irreversible hydrocolloid impression material is also important in producing an accurate impression. The reason for this is that even when set, alginate is a relatively weak material that can easily be distorted or torn. Therefore, the literature reports that a minimum thickness of 3mm is needed when making alginate impressions<sup>1</sup>.

Hydrocolloid impression materials are subject to distortion by several factors. Kendrick described a process by which liquid was lost from impression materials through a process called ‘syneresis.’ This process would then result in a distortion in the details of the impression<sup>5</sup>. The opposite phenomenon by which liquid is absorbed into the impression material is termed ‘imbibition.’ This process results in a swelling of the impression material that also creates a distorted impression.

In 1950, Skinner, Cooper, and Beck<sup>6</sup> tested the dimensional stability of reversible and irreversible hydrocolloid impression materials when stored in 50-60% relative

humidity for 0, 30, and 60 minutes before pouring with dental stone. Their findings were that both reversible and irreversible hydrocolloid impression materials lacked dimensional stability over time and that impressions should be poured immediately after removal from the mouth in order to avoid imbibition/syneresis distortion.

Cohen, *et al.*,<sup>7</sup> also studied the dimensional stability of several alginate products. Impressions were made of an acrylic model of a dental arch. They then measured the accuracy of the casts that were made from the different irreversible hydrocolloid materials after being stored under 5 different conditions before pouring. These conditions were as follows: poured immediately, stored for 10 minutes with a wet paper towel, stored for 30 minutes without a wet paper towel, stored for 1 hour with a wet paper towel and stored for 24 hours with a wet paper towel. Amongst their findings, they concluded that impressions that were poured immediately resulted in the most accurate casts when compared to the original model.

In 1955, Philips<sup>8</sup> published on the physical properties and manipulation of reversible and irreversible hydrocolloid impression materials. He listed five qualities necessary for the accurate reproduction of dental impressions. These are: (1) sufficient fluidity or flow; (2) a gelation time which is adequate to permit injection into the preparation, yet is not unduly prolonged; (3) sufficient strength to resist fracture on removal from the mouth; (4) minimum permanent deformation, and (5) freedom from any deleterious effect on the stone die. The differences and similarities of the two types were presented while the manipulative variables were stressed. Both materials are technique sensitive in the preparation for dental impressions, the making of impressions, and the pouring of dental stones into these impressions. His conclusion was that

impressions must be poured within 15 minutes of removal from the mouth and a fixing solution should be used, if recommended by the manufacturer.

In 1966, the American National Standards Institute and the American Dental Association (ANSI/ADA) established specification No. 18 for alginate impression materials. These specifications were revised in 1997. These specifications include the properties of detail reproduction, recovery from deformation, strain in compression, compressive strength, and gypsum compatibility of irreversible hydrocolloid impression materials<sup>9</sup>.

The method in which an irreversible hydrocolloid impression is removed is also important. In 1969, Rudd, Morrow, and Strunk<sup>10</sup> published a paper describing how to accurately make impressions with alginate. They emphasized that using accurate measurements when mixing is important, but that it was also important to use the proper technique in removing the impression from the mouth with a “firm, quick snap.”

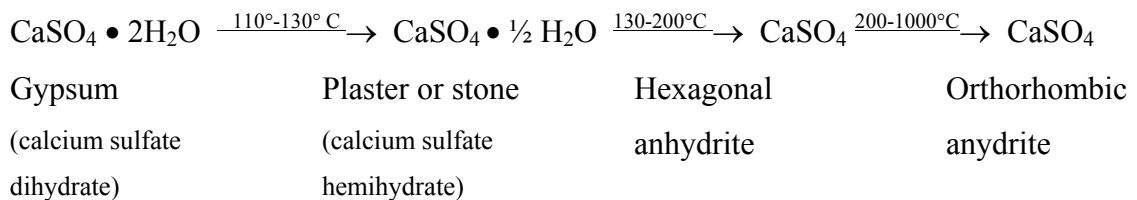
In 1982, Reisbick, Garret, and Smith<sup>11</sup> studied the properties of hand mixed, mechanically mixed, and vacuum-mechanically mixed irreversible hydrocolloid. They found that although there was less air trapped within the samples depending on the mixing techniques, the physical properties of alginate were not affected from a clinical point of view. They also concluded that the method of transferring the impression material from the mixing bowl to the tray was also a possible source of air entrapment.

The ‘alginate alternative’ impression materials that are available on the market are chemically similar to a class of impression materials called “Addition Reaction Silicones”. They are polymerized in a reaction where a platinum salt serves as the catalyst and the vinyl silicone polymer chain has a terminal vinyl group which is then

cross-linked with a hydride group of an adjacent polymer chain.<sup>1</sup> Since the hydrogen atoms are added to the vinyl groups in the reaction, it is thusly named an addition silicone impression material. As long as the proportion of vinyl silicone and hydride silicone are maintained properly and if there are no impurities present, no unwanted byproducts will be created. However, a possible complication of this chemical reaction is that if there is a reaction between the residual hydrides of the base polymer and the moisture in the patient's oral environment, hydrogen gas may develop as a byproduct. This will result in the formation of voids in the gypsum casts if the hydrogen gas is not released from the impression material before being poured. To counteract this possible byproduct formation, manufacturers have added scavengers in the impression materials in the form of noble metals such as platinum or palladium.<sup>1</sup>

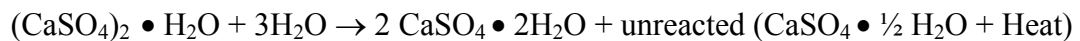
### **Gypsum Products**

The base chemical for dental gypsum is *calcium sulfate dihydrate* ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), which is a mineral deposit that is mined widely throughout the world. The production of dental plaster and stone is achieved by calcining calcium sulfate dihydrate<sup>12</sup>. The gypsum-based product that is used in dental stones and plasters is calcium sulfate hemihydrate,  $\text{CaSO}_4 \cdot \frac{1}{2} \text{H}_2\text{O}$ . The reaction is as follows:



Calcium sulfate hemihydrate is further classified into  $\alpha$  and  $\beta$  forms. The  $\alpha$ -hemihydrate type is characterized by the powder particles being prismatic and more regular in shape than those of the  $\beta$  form. This results in a denser cast with a smoother surface, thus, making them more appropriate for use in dental stones and die stones. On the other hand, the  $\beta$  form is characterized by porous particles that appear to be spongy and irregular in shape. This form of hemihydrate is used in plasters. The difference in surface character and shape of the particles in the  $\alpha$ - and  $\beta$ - forms also account for the amount of water needed in the setting reactions. Due to the porosity and non-uniformity of the  $\beta$  form particles, they will absorb more water, thus resulting in a higher water/powder ration than that of dental gypsum products of the  $\alpha$  form.

The setting reaction of gypsum products is as follows:



The manufacturing process for the different types of dental stones depends on the removal of water and the chemical alteration of  $\alpha$  calcium sulfate hydrates. In order to manufacture a Type III dental stone, water is removed under pressure at 125°C. This leads to the formation of  $\alpha$ -calcium sulfate hemihydrate, thus creating a more uniform shape to the gypsum resulting in a more dense stone. Type IV and V dental stones are produced by taking the  $\alpha$ -calcium sulfate hemihydrate one step further by boiling it in a solution of 30% calcium chloride. This results in crystals being formed in more symmetrical shapes, thus leading to “improved stones” that possess the highest densities of the five types of stones.

In 1969, Rudd et al.<sup>10</sup> published on the procedures for optimal results when mixing gypsum products with water. They recommended that for the best results, premeasured quantities of water should be mixed with predetermined quantities of gypsum products. They also found that the mixing should occur under partial vacuum in a mechanical mixer.

In 1971, Jorgensen and Kono<sup>13</sup> examined the effect of porosity on the compressive strength of dental stone. Dental gypsum products were mixed with the appropriate water/powder ratio and then were mixed mechanically with and without vacuum. These specimens were then examined to calculate their density with an Instron Universal Testing Machine using a load ratio of 1mm per minute. Their findings were that air –bubble porosity was independent of the water to powder ratio and that vacuum-treated stones were denser than the non-vacuum treated. They also found that the compressive strength was increased for those products that were vacuum-treated.

### **Gypsum Compatibility with Irreversible Hydrocolloid**

The fabrication of accurate, high quality casts is an integral part of both the diagnostic and treatment phases of dentistry<sup>10</sup>. Therefore, it is critical that materials that are compatible with one another be used in the fabrication of these casts.

The compatibility of dental gypsum products with irreversible hydrocolloids is a factor that can affect the quality of the casts that are produced when these materials are used in conjunction with one another. There are several reasons affecting this interaction. First, the water in the hydrocolloid impression material acts as a retarder in the setting of gypsum. Secondly, there are fillers, such as borax, in the impression material that can

retard gypsum hardening. The third reason is during the gelation of alginate, sodium sulfate is produced, which in high concentrations is a retarder of gypsum. Many manufacturers have incorporated gypsum hardeners and accelerators, such as sulfate and potassium titanium fluoride, into the irreversible hydrocolloid material to counteract the deficiencies in compatibility.

The compatibility of irreversible hydrocolloid impression materials with gypsum products has been a topic of research. In 1971, Morrow, *et al.*<sup>14</sup> published their findings on the compatibility of five different types of dental stones with alginate impressions of the American Dental Association die. They concluded that Die-Keen (Heraeus Kulzer<sup>TM</sup>, Armonk, NY) was the most compatible gypsum product with the greatest number of alginate impression materials, especially Jeltrate® (Dentsply, York, PA).

In 1973, Owall and Nilner studied the interaction of irreversible hydrocolloid impression material with different brands of dental stones. In order to test this, they fabricated a cone-shaped stainless steel die that had nine 60° screw-like threads/angles. Alginate impressions were made of this die and the seven different brands of stones were then poured into the impressions. Samples were then graded under a microscope to see how accurately they could reproduce the 60° angles. The authors concluded that there was only a slight difference between the compatibility of the alginate impressions with the different brands of dental stones.

In 1980, Jarvis and Earnshaw<sup>15</sup> published a study on the effects of alginate impression materials on gypsum casts. They studied the chemical and physical properties of the alginate-gypsum reaction that could result in the incompatibility of the two materials. The study included five gypsum stones and 10 different irreversible



hydrocolloid impression materials. Both visual examination and scanning electron microscopy (SEM) were used to study the surface characteristics of the specimens. They discovered that calcium sulfate hemihydrate was responsible for some of the incompatibility as it could be found in the casts to the depth of 80 $\mu$ m in poor quality casts. They also found that sodium sulfate could be found in high concentration on the surface of the stone that was considered poor quality. The combination of potassium calcium sulfate (syngenite), unreacted hemihydrate and trace amounts of gypsum were found on the surfaces of the casts that were considered to have the highest quality surface.

In 1981, Jarvis and Earnshaw<sup>16</sup> followed up with an additional article on alginate-gypsum interactions by concentrating on the role of sodium sulfate in incompatibility. Their investigation found that the alginates with the best surface reproduction were those that gave off a high concentration of potassium and sulfate. They suggested that improvements to gypsum compatibility could be made if chemical modifications were made so that a reactor other than calcium sulfate was used. This would eliminate the appearance of sulfate ions in the alginate exudates. A soluble alginate other than sodium alginate and a retarder other than sodium phosphate could also eliminate the presence of sodium ions that decrease the gypsum compatibility of alginates.

In 1983, Carlyle<sup>17</sup> published a study that evaluated the compatibility of 12 different types of irreversible hydrocolloids with three dental stones (Die-Keen, Quickstone, and Hemihydrate). After examining the specimen under (x15) magnification, Die-Keen was found to be the most compatible when rated on a subjective 1-4 scale for reproduction of the 25 $\mu$ m line on the ADA die. No statistically significant

differences were found between the different brands of alginates, including Jeltrate (Dentsply Caulk™).

In 1986, Owen<sup>18</sup> published his study on the compatibility of irreversible hydrocolloid impression materials and dental gypsum. In this study, human saliva was used to lubricate the die to keep the impression material from sticking to the surface, but this did not always achieve the desired effect. He concluded that none of the combinations of impression materials and gypsum were able to reproduce the 25µm line and he had varying results reproducing the 50 and 75µm lines of the standard die.

Also in 1986, Owen<sup>19</sup> published the second part of his study. A new system of grading stone casts attempting to reproduce the 50µm line on the international standard die was described. This system utilized a grading scale of 1-4 that took into consideration the surface quality and the percentage of the length of the 25mm long 50µm wide line.

A score of 1 was defined by the line being reproduced clearly and sharply over the entire length of the 25mm line. This was the best score.

A score of 2 was given when the line was clear over 50% of the length or when the line was indistinct over less than 50%. This line was reproduced over the entire length smoothly but not sharply.

A score of 3 was given if the line was clear over less than 50% of the length and was indistinct over 50% of the length, or if the line was visible over the entire length but was rough or blemished.

A score of 4 was the worst appearance in which the line was not reproduced at all over the entire length. These samples appeared blemished, pitted, rough, etc.

Keuter and Davidson<sup>20</sup> published a study in which they measured the surface roughness of dental stone casts made against alginate impression materials as compared to casts made against an elastomeric impression material. They found that there was greater surface roughness on those casts made against the alginate materials than compared to those made against an elastomeric.

In 1989, Teteruck, *et al*<sup>21</sup> published a study that looked at the quality of 512 gypsum surfaces resulting from 16 gypsums poured against 32 different alginates. These specimens were graded visually and were reported as being superior, average or inferior using photomicrographs for surface roughness. They were also evaluated using a modified Vickers scratch test to assess surface hardness. Their findings were that there was still a wide range of compatibility/incompatibility between the alginates and gypsum products that were available and commonly used in dentistry.

In 1997, Reisbick, *et al*<sup>22</sup> published a study in which they tested three types of alginate materials' compatibility with nine different gypsum materials. This study followed the procedures set forth by ANSI/ADA Specification #18. Their findings were that differences still exist in the compatibility of newly developed gypsum and alginate impression materials.

In 2000, the ANSI/ADA specification #25 was adopted<sup>23</sup>. It is an identical copy of ISO 6873:1998 for dental gypsum products. These specifications standardize the testing and classification of dental gypsum. Classifications of types I-V are based on their setting expansions and compressive strengths.

The ADA/ANSI Specification No. 18 requires that specific types of gypsum products must be tested with the irreversible hydrocolloid impression materials to

determine their compatibility. It requires that one Type III and one Type IV or V gypsum product must be tested with the alginate material.<sup>9</sup> This allows testing in correlation with clinical use since these combinations represent the common use of gypsum with irreversible hydrocolloids by clinician. Specification No. 18 also states that 66% (2/3) of the specimen must be able to reproduce the entire length of the 50µm-wide line (aka, score of “1”) in order to be deemed, “compatible” with the particular gypsum product that is being tested.

In 2002, Heshmati, et al.<sup>24</sup> described the expansion and growth of dental gypsum crystals for up to 120 hours after the initial fabrication of casts. An expansion test unit was used to measure the amount of expansion of casts made with different gypsum products. Die-keen showed the highest degree of setting expansion but was complete at the two-day mark.

### **Studies on Irreversible Hydrocolloid Alternative Materials**

In 1984, Eames and Litvak<sup>25</sup> published on an irreversible hydrocolloid silicone hybrid impression material called Ultrafine (Buffalo Dental Mfg. Co., Brooklyn, NY). Humectants were added to the formulation to help prevent syneresis in hopes of stabilizing the surface detail that could be produced. They found that although the material had a higher tear and compressive strength than traditional alginates, there were no improvements in the dimensional stability.

In 1988, Supowitz, *et al.*<sup>26</sup> tested six different impression materials, including Ultrafine. The authors studied the dimensional accuracy and surface detail of gypsum casts that were made against these impression materials. They concluded that the casts

made from Ultrafine did not have favorable surface quality and that the reference lines were not well defined.

In 2007, Ahmad, *et al.*<sup>27</sup> tested three impression materials for their interaction with a Type III gypsum. The impression materials were a conventional irreversible hydrocolloid, a conventional addition silicone (President PlusJet, Coltene Whaledent AG, Switzerland), and an addition silicone marketed as an alginate alternative (Position Penta, 3M Espe, AG Dental Products Seefeld, Germany). 20 impressions were made of the ADA test die and then stored in distilled water for 10 minutes. Type III gypsum was then poured against these impressions. They found that all of the resultant casts that were poured against these Position Penta impressions were able to reproduce the 50µm line from the test die.

In 2010, Patel *et al.*<sup>28</sup> studied the gypsum compatibility, linear dimensional change and detail reproduction of three irreversible hydrocolloid alternatives. The materials that were tested were Alginot® (Kerr™), Silgimix® (Sultan Dental™), and Position Penta Quick® (3M ESPE™) while Jeltrate Plus antimicrobial® (Dentsply Caulk™) served as the control. These materials were tested according to the ANSI/ADA<sup>28</sup> Specifications 18 and 19. The results were that the test materials had significantly better detail reproduction than the control. All test materials exhibited linear dimensional change of less than 1.0% in accordance with the ADA standard. The gypsum compatibility tests found that Sigimix® was most compatible with Microstone® while Alginot® and Position Penta® exhibited the best compatibility with Die-Keen®. However, an incidental finding was made regarding the deterioration of the Microstone® samples. Although the 50µm lines were discernable at the initial 24-hour mark, when

specimens were examined at a subsequent point in time, changes in detail reproduction in the stone were noted. This also occurred with the Jeltrate®-Die-Keen® samples but to a lesser extent. These serendipitous findings led to the question of whether there was a time affect on gypsum compatibility and led to this research project.

CHAPTER THREE  
MATERIALS AND METHODS

The impression materials that were tested have been marketed as irreversible hydrocolloid (alginate) alternatives. Therefore, the American National Standards Institute and the American Dental Association (ANSI/ADA) Specification No. 18 for Alginate materials was the model for the tests. The parameters for testing are described in the gypsum compatibility model.

**Detail Reproduction**

The impression materials that were used were: (Table 2)

Table 2: Impression materials

<b>CONTROL MATERIAL</b>	<b>MANUFACTURER</b>	<b>LOT #</b>
Jeltrate Plus antimicrobial®	Dentsply Caulk™	081216
<b>TEST MATERIALS</b>		
Silgimix®	Sultan Healthcare™	080815
Position Penta Quick®	3M ESPE™	376972
Alginot	Kerr Corp™	9-1036

Preparation and use of the impression materials were carried out according to the manufacturers' recommendations at room temperature ( $23\pm 2$ )°C. Alginate material was mixed with ( $23\pm 2$ )°C distilled water, using a mechanical mixing bowl (Alginator II®, Cadco™) without reduced atmospheric pressure.

Impressions were made of the American Dental Association die (SABRI Dental Enterprises Inc.™ Lombard, IL) which has three vertical lines: a  $20\mu\text{m}\pm 4$ ,  $50\mu\text{m}\pm 8$ ,  $75\mu\text{m}\pm 8$  and two horizontal lines, both  $75\mu\text{m}\pm 8$ . (See Figures 2,3,4&5) A rigid ring mold was used to support the impression materials during impression making. The ADA ring mold was then placed on a 1/4" glass slab. Impression material was placed into this ring until it was slightly overfilled while seated on the glass. Twenty seconds (20x) before the end of the working time stated by the manufacturer, the clean test block was pressed down into the impression material that was held in the ring mold. The assembly was then immediately placed into a water bath at  $35(\pm 1)$ °C to simulate oral temperature. A second 1/4" glass slab was placed on top and a one kilogram weight provided the pressure to hold the apparatus together. Samples were allowed to set for the manufacturers' stated setting time plus an additional three minutes.

The Jeltrate Plus antimicrobial was mixed using 25grams of powder with 57mls of distilled water as recommended by the manufacturer. Distilled water at  $23 (\pm 1)$ °C was combined with the powder in a mechanical mixing bowl (Alginator) for 30 seconds to simulate clinical preparation.

The test materials were distributed in automix cartridges. After extruding a small amount of the base and catalyst pastes from the cartridges to ensure adequate flow, automix tips were used in dispensing these materials.



Between impressions of the ADA die, careful cleaning with steam was carried out. The die was then allowed to dry for 1 minute. Saliva was manually applied to the die as a lubricant before making impression with irreversible hydrocolloid.

The working and setting time for the tested impression materials are outlined as follows: (Table 3)

Table 3: Working and Setting times for impression materials

<b>IMPRESSION MATERIAL</b>	<b>WORKING TIME</b> (Minutes:Seconds)	<b>SETTING TIME</b> (Minutes:Seconds)
*(Control)	2:15	4:00
Jeltrate Plus Antimicrobial®		
Alginot®	1:00	2:30
Silgimix®	1:00	2:10
Position Penta Quick®	1:00	2:40

The specimen was then removed from the water bath, the ring mold separated from the die (see Figure 6) and the sample was inspected immediately using low angle magnification 10x (Leica Zoom 2000® Leica Microsystems GmbH™, Wetzlar, Germany). Only the impressions that reproduce the entire 25mm length of the 50µm±8 line were used for the gypsum compatibility test. The specimens were then graded on a scale of 1-4 as described by Owen in 1986. (See Figure 6)

The grading is outlined as follows:

A score of “1” was defined by the line being reproduced clearly and sharply over the entire 25mm length of the 57 $\mu$ m-wide line. This was the best score and the score accepted as “gypsum compatible”.

A score of “2” was given when the line was clear over 50% of the length or when the line was indistinct over less than 50%. This line was reproduced over the entire length smoothly but not sharply.

A score of “3” was given if the line was clear over less than 50% of the length and was indistinct over 50% of the length, or if the line was visible over the entire length but was rough or blemished.

A score of “4” was the worst appearance in which the line was not reproduced at all over the entire length. These samples appeared blemished, pitted, rough, etc.

### **Gypsum Compatibility**

After inspection and grading of the test specimen, only those that had reproduced the entire length of the 50 $\mu$ m-wide line were used for the gypsum compatibility test.

Following the impressions of the ADA die with the different materials, a number of gypsum products were used with each of the impression materials to fabricate casts. The gypsum products that were tested represented Types III, IV, and V of gypsum as defined by the ANSI/ADA Specification #25. These included the following gypsum products: (Table 4)

Table 4: Dental Gypsum Products

<b>ANSI/ADA CLASSIFICATION</b>	<b>GYP SUM MATERIAL</b>	<b>MANUFACTURER</b>	<b>LOT #</b>
Type III	Microstone Golden®	Whip Mix Corp. <sup>TM</sup> (Louisville, KY)	027011002
Type IV	Prima-Rock®	Whip Mix Corp. <sup>TM</sup> (Louisville, KY)	59090602
Type V	Die-keen Green®	Heraeus Kulzer <sup>TM</sup> (Armonk, NY)	1002033

These gypsum products were mixed in accordance with the manufacturers’ directions on water-powder ratios and mixing times. A graduated cylinder was used to measure the required volume of distilled water 23 (+/-1)°C. The water was added to the mixing bowl first and then the appropriate amount of powder was added. Hand mixing of the samples was carried out for 15 seconds to ensure proper wetting and initial mix. All gypsum products were then vacuum mixed (20mmHg) in a Whip Mix Combination Unit for the time period recommended by the manufacturer. A separate mixing bowl, mechanical spatula, and manual spatula were dedicated for each different type of gypsum product and only those mixing apparatuses were used with their respective products. The working and setting times for each of the tested gypsum products are: (Table 5)

Table 5: Working and Setting times for dental gypsum products tested

<b>GYPSUM</b>	<b>POWDER:WATER RATIO</b> (Grams:mls)	<b>MIXING TIME</b> (Seconds)	<b>SETTING TIME</b> (Minutes)
Microstone Golden ®	140:40	20-30	13
Prima-Rock®	100:20	20-30	12
Die-Keen Green®	60:13	20-30	13

A thin layer of vacuum mixed gypsum was then vibrated into the slit mold containing the accepted impressions. The rest of the mold was then filled and these specimens were allowed to set for their respective manufacturers' recommended setting times plus an additional 30 minutes. The slit mold containing the gypsum cast was then separated from the ring mold containing the impression material. The casts were marked on the underside to identify the impression material and sample number. Examinations of the specimen were accomplished with the aid of a light microscope at 20-degree incident surface lighting and at 10x magnification (Leica Zoom 2000® Leica Microsystems GmbH™, Wetzlar, Germany). Surface characteristics were then graded subjectively on a scale of 1-4 as outlined by Owen in 1986.

The effect of time on the incompatibility of some gypsum products that was observed by Patel, *et al*<sup>28</sup>, was also studied. A pilot study was conducted to verify which time points were the most appropriate for examination of specimens. Initial grading was carried out at the time of initial separation of the gypsum casts from the impressions (Immediate/baseline). Additional grading was carried out at the 24-hour (1 day), 48-hour

(2 day), 168-hour(7 day) and 336-hour (14 day) time points. The scores were then analyzed to determine if there was a time effect on the interaction between the different gypsum products and impression materials.

An HX85 Hygrometer® (Omega Engineering, Inc. Stamford, CT) was used to monitor the relative humidity of the laboratory in conjunction with its monitoring software (Omega USB Products. Version 1.00.09.309).

Each of the parameters that were tested consisted of 10 samples of each material group. All specimens were fabricated and examined by the author.

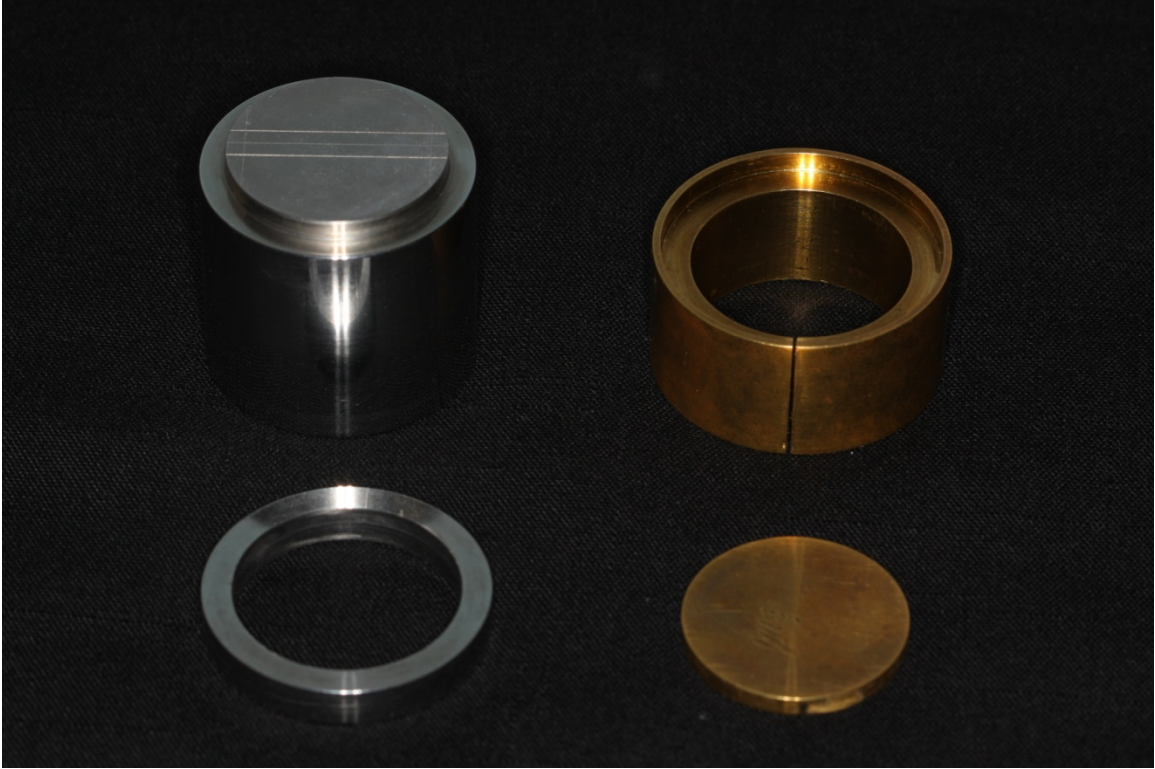


Figure 2: The ANSI/ADA Specification No. 18 Testing Apparatus  
From upper left going clockwise:

- A. Test die
- B. Brass slit mold
- C. Brass plate
- D. Test mold



Figure 3: Surface of the ANSI/ADA Specification No. 18 die

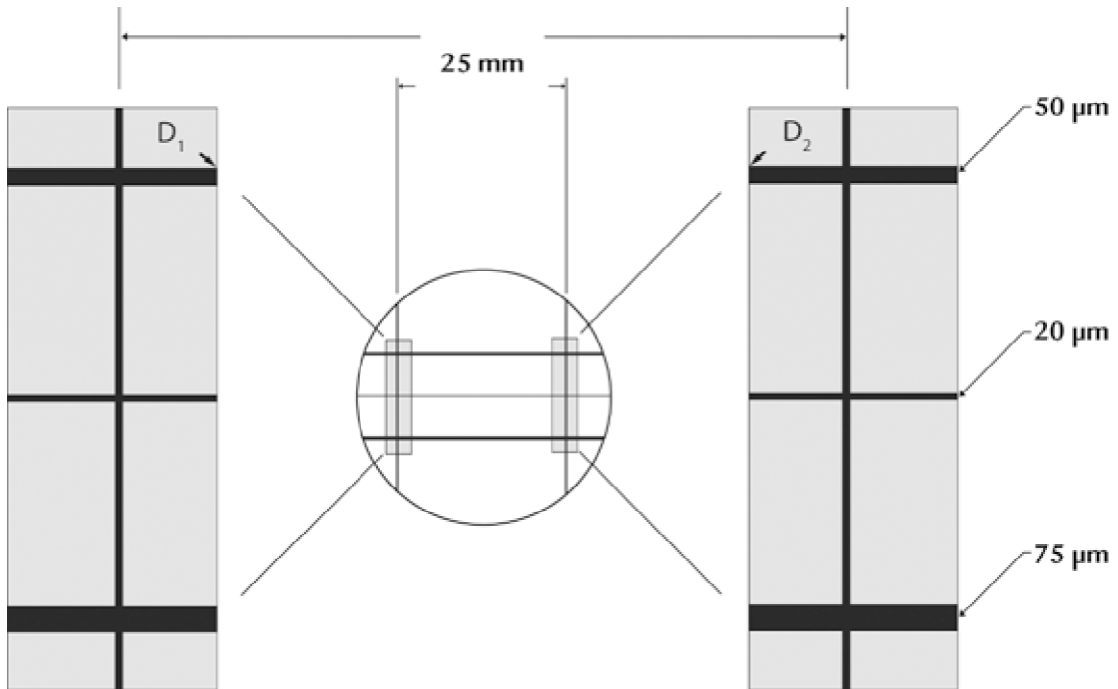


Figure 4: Schematic drawing of the dimensions of the ANSI/ADA Specification No. 18 die surface.



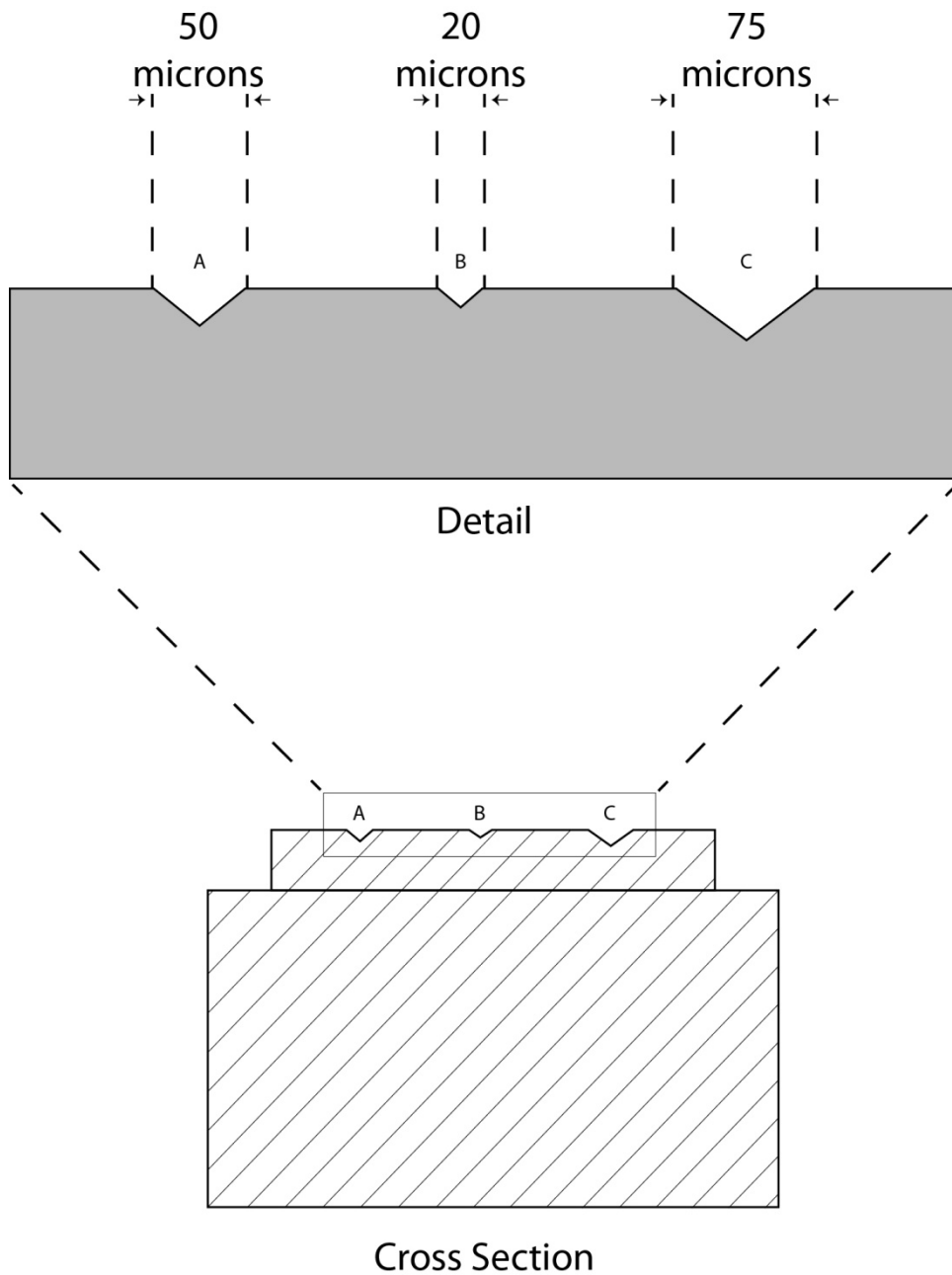


Figure 5: Schematic of the ANSI/ADA Specification No. 18 die surface from a lateral view.

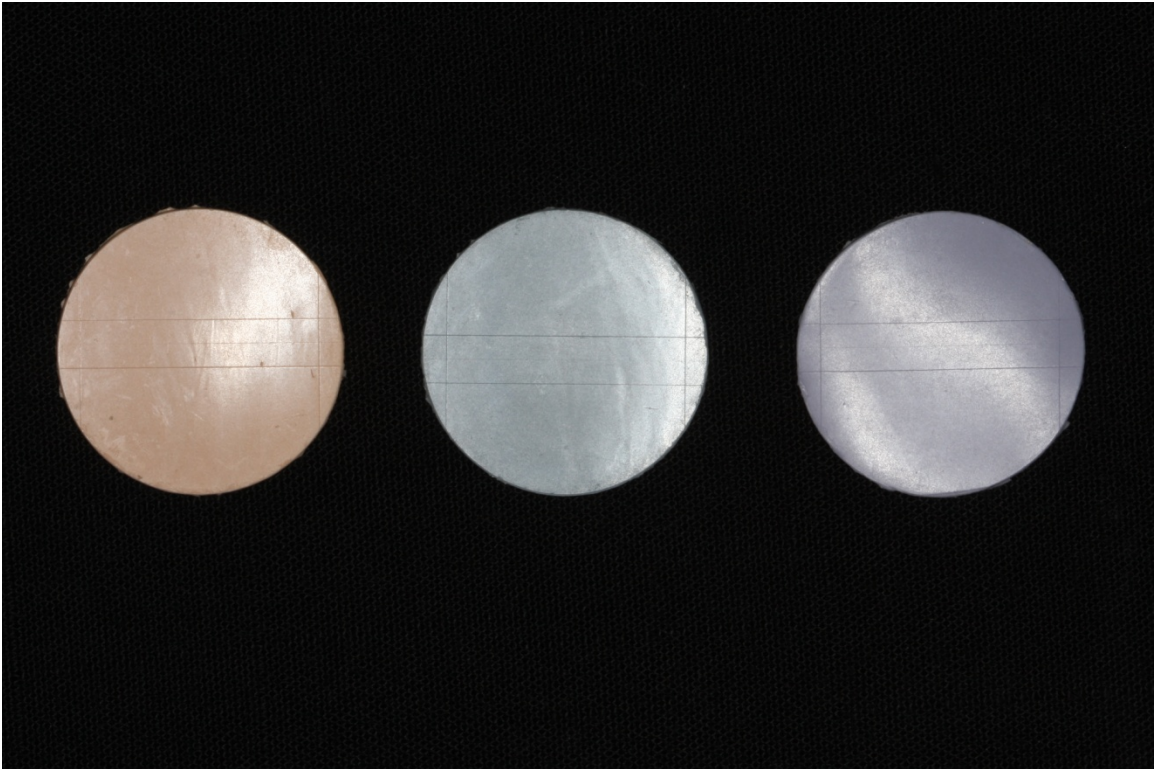


Figure 6: Test impression materials

From left to right:

- A. Alginot®
- B. Silgimix®
- C. Position Penta Quick®

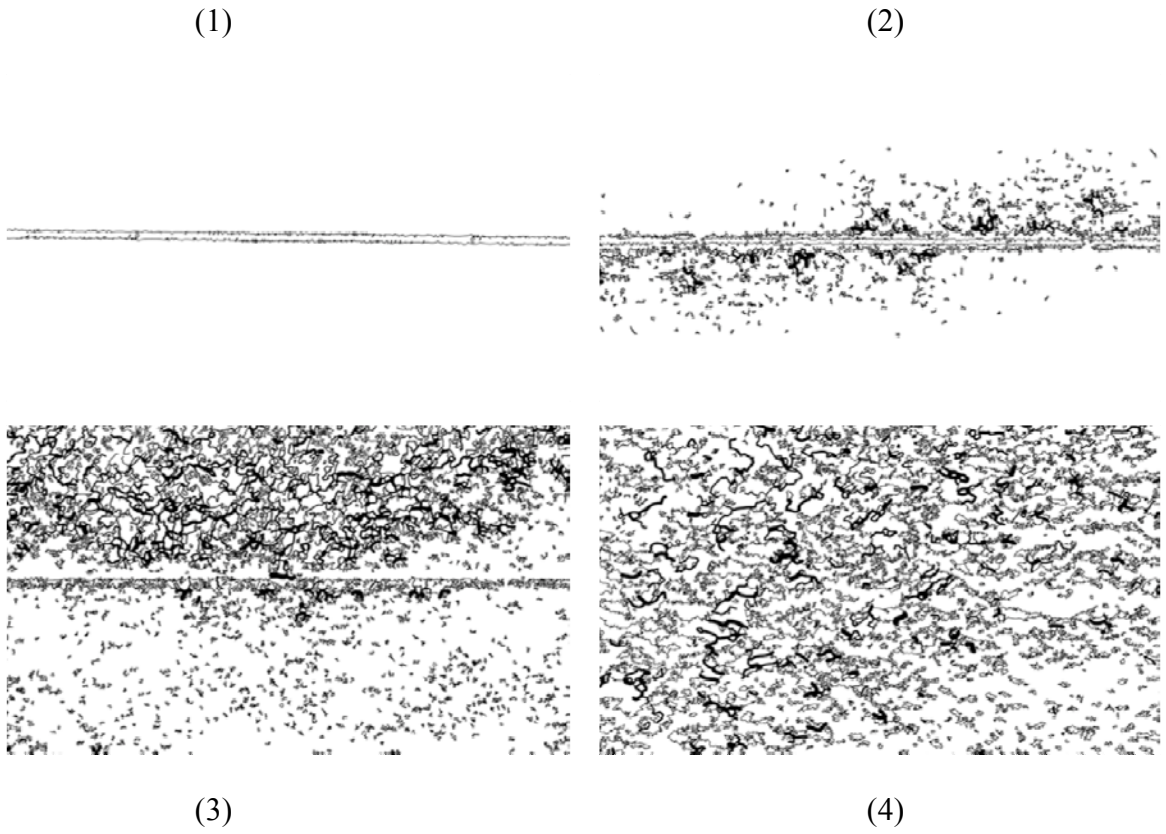


Figure 7: Schematic representing the grading scale of samples as described by Owen.<sup>18</sup>

## CHAPTER FOUR

### RESULTS

The guidelines of ANSI/ADA Specification #18 were used in analyzing all specimens.

The detail reproduction grading criteria described by Owen in 1986<sup>18</sup> was facilitated. This scoring system was based on the impression materials' ability to reproduce the entire 25mm length of the 50 $\mu$ m-wide line. All the impression materials that were tested were able to reproduce the aforementioned line for each of the 40 samples that were created, as this was the requirement by the parameters of this study before the impressions could be poured with the different types of gypsum products. The ordinal scoring system was:

A score of "1" was defined by the line being reproduced clearly and sharply over the entire 25mm length of the 57 $\mu$ m-wide line. This was the best score and the one score accepted to represent "gypsum compatibility".

A score of "2" was given when the line was clear over 50% of the length or when the line was indistinct over less than 50%. This line was reproduced over the entire length smoothly but not sharply.

A score of "3" was given if the line was clear over less than 50% of the length and was indistinct over 50% of the length, or if the line was visible over the entire length but was rough or blemished.

A score of “4” was the worst appearance in which the line was not reproduced at all over the entire length. These samples appeared blemished, pitted, rough, etc.

All impression materials (Alginate and Alginate Alternatives) were able to attain a score of “1”. This resulted in 10 different specimens for each impression material, all with the ability to clearly and sharply reproduce the entire length of 50µm-wide line.

### **Gypsum Compatibility**

The ANSI/ADA Specification No. 18 was used to grade the gypsum models that were created by pouring the impressions that were made against the ADA die.

Specification No. 18 describes that 66% (2/3) of the specimen must be able to reproduce the entire length of the 50µm-wide line (aka, score of “1”). Examinations of the specimen were accomplished with the aide of a light microscope at 20-degree incident surface lighting and at 10x magnification (Leica Zoom 2000® Leica Microsystems GmbH™, Wetzlar, Germany). The grading of the specimen was carried out immediately after separation from the impressions (baseline), 24hrs, 48hrs, 168hrs, and 336hrs after separation.

### **Statistical Analysis**

The ADA die served as the experimental unit for this research and three different factors [A (fixed), B (fixed) and C (within)] were tested. “Factor A” consisted of the three (3) different gypsum products, “Factor B” was the four (4) different impression materials, and “Factor C” consisted of the five (5) different time intervals ( $t_1, t_2, t_3, t_4, t_5$ ) at which the specimen were examined. Therefore, there were a total of 60 different treatment combinations ( $3 \times 4 \times 5 = 60$ ). A total of ten (10) samples of each treatment

combination were tested, resulting in a total of 600 sample recordings. The scoring system that was facilitated was on an ordinal scale and was non-parametric in nature. The “family-wise” Three-Factor ANOVA test was facilitated to analyze the interactions between the experimental pairings and how these interactions compared among different time points. A significance level of  $\alpha = 0.05$  was used.

### Test Group

After data collection, all the treatment combinations among the alternative materials and gypsum achieved a score of “1” at baseline and remained constant at all other time points. Alternatively, there were differences, some of which were statistically significant, in scores for the treatment combinations between the irreversible hydrocolloid impression material (Jeltrate Plus antimicrobial®) and the different gypsum products at different time points.

### Control Group

When examining the score for the specimens that were made with the control impression material, there was both a difference in scores related to stone and time. Among the gypsum products tested with the control, it was found that significantly better scores were recorded with the Type V gypsum (Die Keen®) than those recorded with the Type III (Microstone®) [ $p < .001$ ] or Type IV (Prima Rock®) [ $p = 0.032$ ].

Comparisons between Control (alginate) and Type V gypsum (Die Keen®) among the different points in time was analyzed via the 3-factor ANOVA test. All time periods after 24hrs demonstrated significantly better reproducibility ( $p = 0.01$ ) than when

the specimens were evaluated at earlier time points ( $\leq 24$ hrs). No other statistically significant differences among the time points were observed.

According to the ANSI Specification No. 18's gypsum compatibility test, greater than 66% (2/3) of the tested samples must be able to reproduce the 25mm length of the 50 $\mu$ m-wide line. This means that a score of "1" must be attained. According to the findings in this study, we see that all the test groups (Alginate Alternatives) tested "compatible" by being able to reproduce the line at all time periods with all the gypsum products that were tested. (Figure 8)

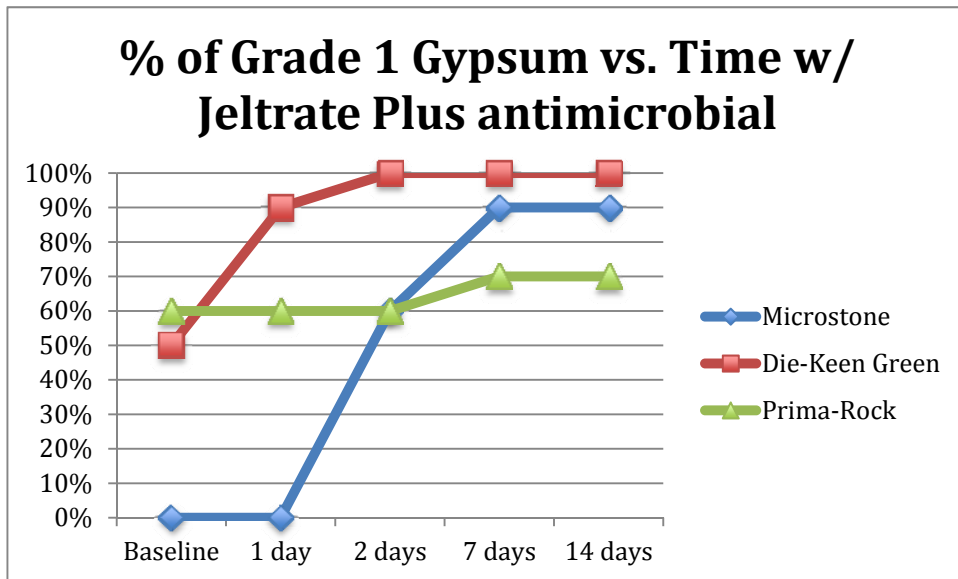


Figure 8: Graph demonstrating the relationship between time points and the percentage of scores of "1"

The control group showed varied results for gypsum compatibility. The Alginate and type III gypsum (Microstone®) combination was “incompatible” at baseline after separation from the impression, with none (0/10) of the specimen being graded as “1”. At the 24hr time point, there was an improvement in scores but still none were graded as “1”. At 48hrs, 60% (6/10) samples were graded as “1”. At the 168 and 336hrs time points, 90% (9/10) of the samples were graded as a “1”, thus, displaying “compatibility” at these time points.

The Alginate and Type IV gypsum (PrimaRock®) group showed a score of “1” for 60% (6/10) of samples immediately after separation, at 24 and 48hrs. Then, at the 168 and 336hr time points, the samples were graded with a score of “1” for 70% (7/10), thus deeming them “compatible” at these time points. It was this particular combination that reached “compatibility” status at the latest time point.

The Alginate and Type V gypsum (DieKeen®) group showed a “1” score in 50% (5/10) of samples immediately after separation. However, at 24hrs, 90% (9/10) of samples were scored at “1”. 100% (10/10) of the samples attained a score of “1” by the 48-hr mark and these values remained stable for the remaining time periods.

When comparing the time effect on the gypsum compatibility of Alginate and type III gypsum (Microstone®), it was seen that “compatibility” status was reached between the time points of 48hrs (Day 2) and 168hrs (Day 7). In order to analyze this change, McNemar’s test was used to test between the time points of 48hrs and 168hrs. (Table 6)



Table 6: Table demonstrating McNemar’s test to determine changes in scores between time points

		<b>168 HRS/DAY 7</b>	
		<b>Grade 1</b>	<b>Grade 2</b>
<b>48HRS/DAY 2</b>	<b>Grade 1</b>	6	0
	<b>Grade 2</b>	3	1

Although the results of McNemar’s test did not result in a statistically significant difference ( $p= 0.0833$ ), there was a clinically significant difference that resulted in a change from 60% to 90% of the casts being scored as a “1”, resulting in a change from “incompatible” to “compatible.”

### **Summary of Results**

We conclude from the results of statistical analysis that we accept our alternative hypothesis. There is a significant difference in gypsum compatibility between irreversible hydrocolloid and the alternative impression materials. There is also a change in the gypsum casts at different time points when used in combination with the irreversible hydrocolloid impression materials.

The null hypothesis is rejected, that there is no significant difference in gypsum and that there is no change in the gypsum casts at different time points. There does exist an interaction with the alginate alternatives, gypsum products and time when compared to the alginate, gypsum products and time. Overall, there was an increase in the surface quality of gypsum products as time was increased.

## CHAPTER FIVE

### DISCUSSION

Irreversible hydrocolloid impression materials are currently used in dentistry in a wide range of applications. Some of these uses include the fabrication of casts for diagnostic purposes, the construction of removable dental prostheses and for the creation of opposing casts in the fabrication of fixed dental prostheses. Its availability on the market; relatively low cost and ease of handling are factors that have contributed to its popularity.

A number of “alginate alternative” materials are currently available on the market. These impression materials can be classified as “elastomeric” in nature but have been marketed and compared to the irreversible hydrocolloids. There have been a number of reports in the dental literature that have demonstrated the detail reproduction ability of poly (vinyl siloxane) impression materials, in accordance with the ANSI/ADA specification No. 19 – Dental Elastomeric Impression Materials.<sup>29</sup> Chee and Donovan<sup>30</sup> as well as Ragain, et al.<sup>31</sup> tested poly (vinyl siloxane) materials and found that they passed the test of being able to consistently reproduce the 20µm line of the ADA die. In the current study, due to the manufacturers’ claims for the test impression materials being “Alginate (irreversible hydrocolloid) Alternatives”, the ANSI/ADA specification No. 18 for Alginate impression materials was used.

Patel *et al.*<sup>28</sup> found that there was a statistically significant difference between the irreversible hydrocolloid and the three test Alginate Alternatives that were examined in

the current study. They found that the Alginate Alternatives were superior in their ability to reproduce the 50µm line. However, there were no clinically or statistically significant differences between the different alginate alternative impression materials, as was confirmed in the current study.

### **Gypsum Compatibility Test**

Three different types of dental gypsum products representing three different classes of stones that are commonly used for fixed and removable prosthodontics were tested. These dental stones are classified according to the ANSI/ADA Specification No. 25 –Dental Gypsum Products.<sup>23</sup> The Type III stone (Microstone Golden®) is commonly used in the fabrication of master casts in removable prosthodontics and for opposing casts in fixed prosthodontics. It is characterized by high strength and low expansion and it is a common practice to use this stone in combination with irreversible hydrocolloids, due to its relative low cost.

Type IV stones (e.g. PrimaRock®) are characterized by high strength and low expansion, leading to a more accurate cast. These stones are commonly used for casts/dies in fixed prosthodontic procedures when the accurate fit of the prosthesis is most desired.<sup>12</sup>

Type V stones (e.g. Die-Keen Green®) are characterized by high strength and high expansion. They are designed for use in the fabrication of dies for fixed prosthodontic procedures (crowns, fixed partial dentures, etc) when expansion of the stone is desired to compensate for dimensional changes during dental casting procedures.<sup>12, 32</sup>

The current study confirmed the findings of Morrow, *et al.*<sup>14</sup> who reported Die-Keen® was the most compatible gypsum product with Jeltrate® (Dentsply, York, PA). These findings were also confirmed by Carlyle<sup>17</sup> in a study that evaluated the compatibility of 12 different types of irreversible hydrocolloids with three dental stones, including Die-Keen®. No statistically significant differences were found between the different brands of alginates, including Jeltrate® (Dentsply Caulk™).

A number of studies have been published on the causes of incompatibility between irreversible hydrocolloid impression materials and dental gypsum products. Jarvis and Earnshaw<sup>15</sup> studied the chemical and physical properties of the alginate-gypsum reaction by both visual examination and scanning electron microscopy (SEM) of the surface characteristics of gypsum casts that had been poured against irreversible hydrocolloids. They discovered that for the stone that was considered poor quality, calcium sulfate hemihydrate, which is instrumental in forming the alginate gel, was found to the depth of 80µm. Sodium sulfate, which acts as a retarder, was found in high concentration on the surface of poor quality casts. (Table 1) The combination of potassium calcium sulfate (syngenite), unreacted hemihydrate and trace amounts of gypsum were found on surfaces of casts considered to have the highest quality.

Jarvis and Earnshaw<sup>16</sup> studied the role of sodium sulfate in incompatibility. Their investigation found that the alginates with the best surface reproduction were those that gave off a high concentration of potassium and sulfate. They suggested that improvements to gypsum compatibility could be made if chemical modifications were made so that a reactor other than calcium sulfate was used. This would eliminate the appearance of sulfate ions in the alginate exudates. A soluble alginate other than sodium

alginate and a retarder other than sodium phosphate could also eliminate the presence of sodium ions that decrease the gypsum compatibility of alginates.

The present study was limited in its evaluation of the quality of the gypsum casts in that a subjective, visual scale was used to grade the specimens.<sup>18</sup> No attempt was made to determine the chemical composition of the gypsum surface. Therefore, it is not possible to determine what the cause of the changes in surface quality at different time points could be attributed to. One can speculate that it could be due to a continued reaction of the chemicals that are a byproduct of the alginate setting reaction, the dental gypsum setting reaction or interactions between the byproducts of the two reactions.

Heshmati, et al.<sup>24</sup> described the expansion and growth of dental gypsum crystals for up to 120 hours after the initial fabrication of casts. Die-keen showed the highest degree of setting expansion but was complete at the two-day mark.

Another possible cause for the observed changes in the gypsum casts could be attributed to the amount of water that is in the casts. The amount of water in each type of stone decreases from type III, IV to V stone with Microstone® having the highest water-powder ratio and Die-Keen® having the lowest. Through the course of data collection, the most dramatic change in scores was seen within the Microstone® group, which could possibly be explained by the fact that these casts had the most water to lose. The Die-Keen® group had the least amount of water to lose and resulted in the greatest percentage of its casts attaining scores of “1”.

Due to the fact that gypsum compatibility over time had not been previously reported in the literature, the storage conditions of the samples were not defined in the ANSI/ADA Specification No. 18. An HX85 Hygrometer® (Omega Engineering, Inc.

Stamford, CT) was used to monitor the relative humidity of the laboratory in conjunction with its monitoring software (Omega USB Products. Version 1.00.09.309). Relative humidity ranged between 50.5% to 65.0% during the two weeks of testing. However, this seemed to be within normal limits of the laboratory setting.

A pilot study helped determine the different time points for analysis. Time points were generally based on the common protocol of dental clinicians and laboratory technicians. The immediate/baseline grading was done in accordance with the ANSI/ADA Specification No. 18. The pilot study helped identify that there had been dynamic changes in the gypsum casts between baseline and 48-hrs. Then, it was assumed, from anecdotal observations, that alginate impressions would be poured at the dental office and then sent to a dental laboratory. It was then assumed that since most laboratories have approximately a two-week turn around, the casts would sit for several days in the lab before being worked on. Thus, the 168hr/7-day time point was chosen and 336hrs was the point in time when most casts/laboratory work would be completed.

The “family-wise” Three-Factor ANOVA test was facilitated to analyze the interactions between the experimental pairings and how these interactions compared between different time points. The results showed that all of the Alginate Alternative impression materials were able to achieve a score of “1” and the gypsum casts that were made from these impressions recorded a score of “1” at all the measured time points. This shows that the Alginate Alternatives demonstrate an equally high degree of gypsum compatibility over time. However, the variance in scores was recorded within the Control group with different combinations of irreversible hydrocolloid and gypsum attaining “Gypsum Compatibility” status (66%) at different points in time. Within the

Control Group, Die-Keen® demonstrated the ability to reach the 66% mark by the 24-hour time point. Microstone® and Prima-Rock® displayed a slower rate of attaining compatibility at some point between the two-day and seven-day time points. However, it is notable that a higher percentage of Microstone® (90%) versus Prima-Rock® (70%) casts were able to attain compatibility. Although these differences were not statistically significant, there are clinically significant implications to these findings. It can be hypothesized that casts made from the alginate-Microstone® combination might not be recommended for use in the fabrication of prostheses that require a high degree of precision, such as removable partial denture frameworks. Furthermore, if this combination is used, a waiting period of two to seven days may be necessary to improve accuracy of the casts.

One of the limitations of the current study was that it had limited application to clinical dental procedures. Although saliva was used to lubricate the ADA die and the temperature mimicked oral temperatures, there are other chemicals/substances that can be present in the oral environment. Furthermore, it is common practice to disinfect the impressions before pouring them, which was not done in this study. Various authors have published on the effects disinfection has on both alginate and elastomeric impression materials.<sup>33, 34</sup>

Overall, there was an increase in the surface quality of gypsum products. However, it is not known what would happen to these surfaces in a clinical/laboratory setting once they were handled for trimming, mounting and manipulation during different procedures. It is a common practice for clinicians to trim gypsum casts with a mechanical lathe that uses water to wash away the slurry that is created from the trimmed

gypsum material. It is the investigator's opinion that further study should be done in order to determine the affect of additional water being added to the stone during trimming procedures.



## CHAPTER SIX

### CONCLUSIONS AND FUTURE DIRECTIONS

This study investigated the gypsum compatibility over time of a number of irreversible hydrocolloid impression material alternatives. Gypsum compatibility was tested according to the ANSI/ADA Specification No. 18 – Alginate Impression Materials. Analysis was conducted using a visual scoring system of 1 to 4. Ten impressions of the ADA die were made with each material (one irreversible hydrocolloid and three alternatives) for the gypsum compatibility tests. For each impression material, ten samples were poured with Type III (Microstone Golden®), ten with Type IV (Prima-Rock®) and ten with Type V gypsum (Die-Keen Green®). Specimens were analyzed at baseline, 24 hours, 48 hours, 168 hours (7days), and 336 hours (14 days) using a microscope.

Within the limitations of the current study, the following conclusions can be made:

1. All of the gypsum casts made with the “Alginate Alternative” impression materials demonstrated gypsum compatibility at all time points without change.
2. All combinations of irreversible hydrocolloid (Jeltrate Plus antimicrobial®) and dental gypsum products demonstrate initial incompatibility but then showed differences in compatibility at different points in time and between the different combinations.

3. Jeltrate Plus antimicrobial® and Microstone Golden® demonstrated initial incompatibility but changed over time to be compatible by the 168hr (7 day) point, after which no further change was observed.
4. Jeltrate Plus antimicrobial® and Die-Keen Green® combination also demonstrated initial incompatibility at baseline but then became compatible by the 24 hour time point and detail reproduction was enhanced up to 48 hours, after which no further change was observed.
5. Jeltrate Plus antimicrobial® and Prima-Rock® also started out as incompatible and showed the least amount of change in the casts, eventually reaching compatibility levels by 168hrs (7 days), after which no further change was observed.
6. Clinical implications of these findings are that casts made from the alginate-Microstone® combination might not be recommended for use in the fabrication of prostheses that require a high degree of precision, such as removable partial denture frameworks. If this combination is used, a waiting period of two to seven days may be necessary to improve accuracy of the casts.
7. There is a significant difference within the Jeltrate Plus antimicrobial® (control) group at all time points before and after 24 hours.
8. The null hypothesis is rejected because there is a significant difference between the Alginate Alternatives and the Irreversible Hydrocolloid impression material. Time has an effect on the compatibility of irreversible hydrocolloid impression materials and dental gypsum.

## REFERENCES

1. Shen C. Impression Materials. In: Anusavice KJ, (ed.). *Phillips' Science of Dental Materials 11th Edition*. Elsevier, St. Louis, Missouri, 2003, p. 205-42.
2. Buchan S and Peggie RW. Role of ingredients in alginate impression compounds. *J Dent Res*. 1966; 45: 1120-9.
3. Skinner EW, and Pomes, C.E. Dimensional Stability of Alginate Impression Materials. *Journal of the American Dental Association*. 1946; 33:1253.
4. Skinner EW, and Pomes, C.E. Alginate Impression Materials: Technic for Manipulation and Criteria for Selection. *The Journal of the American Dental Association*. 1947; 34:245.
5. Kendrick ZV, Jr. The physical properties of agar type hydrocolloid impression material. *J Am Dent Assoc*. 1950; 40: 575-84.
6. Skinner EW, Cooper EN and Beck FE. Reversible and irreversible hydrocolloid impression materials. *J Am Dent Assoc*. 1950; 40: 196-207.
7. Cohen BI, Pagnillo M, Deutsch AS and Musikant BL. Dimensional accuracy of three different alginate impression materials. *J Prosthodont*. 1995; 4: 195-9.
8. Phillips RW. Physical properties and manipulation of reversible and irreversible hydrocolloid. *J Am Dent Assoc*. 1955; 51: 566-72.
9. American National Standards Institute/American Dental Association Council of Scientific Affairs. Chicago I. American Dental Association Specification No. 18. Alginate Impression Materials. 1997.
10. Rudd KD, Morrow RM and Strunk RR. Accurate alginate impressions. *J Prosthet Dent*. 1969; 22: 294-300.
11. Reisbick MH, Garrett R and Smith DD. Some effects of device versus handmixing of irreversible hydrocolloids. *J Prosthet Dent*. 1982; 47: 92-4.
12. Anusavice KJ. Gypsum Products. In: Anusavice KJ, (ed.). *Phillips' Science of Dental Materials, 11/e*. 11 ed. St Louis, Missouri: Elsevier Science (USA), 2003, p. 255-81.

13. Jorgensen KD and Kono A. Relationship between the porosity and compressive strength of dental stone. *Acta Odontol Scand.* 1971; 29: 439-47.
14. Morrow RM, Brown CE, Jr., Stansbury BE, DeLorimier JA, Powell JM and Rudd KD. Compatibility of alginate impression materials and dental stones. *J Prosthet Dent.* 1971; 25: 556-66.
15. Jarvis RG and Earnshaw R. The effects of alginate impressions on the surface of cast gypsum. I. The physical and chemical structure of the cast surface. *Aust Dent J.* 1980; 25: 349-56.
16. Jarvis RG and Earnshaw R. The effect of alginate impressions on the surface of cast gypsum. II. The role of sodium sulphate in incompatibility. *Aust Dent J.* 1981; 26: 12-7.
17. Carlyle LW, 3rd. Compatibility of irreversible hydrocolloid impression materials with dental stones. *J Prosthet Dent.* 1983; 49: 434-7.
18. Owen CP. An investigation into the compatibility of some irreversible hydrocolloid impression materials and dental gypsum products. Part II. A refined discriminatory procedure. *J Oral Rehabil.* 1986; 13: 147-62.
19. Owen CP. An investigation into the compatibility of some irreversible hydrocolloid impression materials and dental gypsum products. Part I. Capacity to record grooves on the international standard die. *J Oral Rehabil.* 1986; 13: 93-103.
20. Keuter FM and Davidson CL. Surface roughness of dental stone casts from alginate impressions. *J Dent.* 1986; 14: 23-8.
21. Teteruck WR, Johnson LN, Mills AR and Downar-Zapolski B. Surface quality of gypsums when poured against alginates. *J Can Dent Assoc.* 1989; 55: 545-9.
22. Reisbick MH, Johnston WM and Rashid RG. Irreversible hydrocolloid and gypsum interactions. *Int J Prosthodont.* 1997; 10: 7-13.
23. American National Standards Institute/American Dental Association Council of Scientific Affairs. Chicago I. American Dental Association Specification No. 25. Dental Gypsum Products. . 2000.
24. Heshmati RH, Nagy WW, Wirth CG and Dhuru VB. Delayed linear expansion of improved dental stone. *J Prosthet Dent.* 2002; 88: 26-31.
25. Eames WB and Litvak CS. New irreversible hydrocolloid silicone impression material. *J Prosthet Dent.* 1984; 52: 479-84.

26. Supowitz ML, Schnell RJ, Dykema RW and Goodacre CJ. Dimensional accuracy of combined reversible and irreversible hydrocolloid impression materials. *J Prosthet Dent*. 1988; 59: 404-9.
27. Ahmad S, Tredwin CJ, Nesbit M and Moles DR. Effect of immersion disinfection with Perform-ID on alginate, an alginate alternative, an addition-cured silicone and resultant type III gypsum casts. *Br Dent J*. 2007; 202: E1; discussion 36-7.
28. Patel RD, Kattadiyil MT, Goodacre CJ and Winer MS. An in vitro investigation into the physical properties of irreversible hydrocolloid alternatives. *J Prosthet Dent*. 2010; 104: 325-32.
29. American National Standards Institute/American Dental Association Council of Scientific Affairs. Chicago I. ANSI/ADA Specification No. 19 - Dental Elastomeric Impression Materials. 2004.
30. Chee WW and Donovan TE. Polyvinyl siloxane impression materials: a review of properties and techniques. *J Prosthet Dent*. 1992; 68: 728-32.
31. Ragain JC, Grosko ML, Raj M, Ryan TN and Johnston WM. Detail reproduction, contact angles, and die hardness of elastomeric impression and gypsum die material combinations. *Int J Prosthodont*. 2000; 13: 214-20.
32. Anusavice KJ. Casting Investments and Procedures. In: Kenneth J. Anusavice P, DMD, (ed.). *Phillips' Science of Dental Materials*. Eleventh Edition ed. St. Louis, Missouri (USA): Elsevier Science (USA), 2003, p. 295-350.
33. Tobias RS, Browne RM and Wilson CA. An in vitro study of the antibacterial and antifungal properties of an irreversible hydrocolloid impression material impregnated with disinfectant. *J Prosthet Dent*. 1989; 62: 601-5.
34. Johnson GH, Chellis KD, Gordon GE and Lepe X. Dimensional stability and detail reproduction of irreversible hydrocolloid and elastomeric impressions disinfected by immersion. *J Prosthet Dent*. 1998; 79: 446-53.

APPENDIX A

CONTROL DATA

This table shows the gypsum compatibility scores for the Control and Type III dental gypsum (Microstone Golden®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	3	2	2	1	1
2	3	2	2	1	1
3	3	3	2	1	1
4	2	2	1	1	1
5	2	2	1	1	1
6	3	3	2	2	2
7	2	2	1	1	1
8	2	2	1	1	1
9	2	2	1	1	1
10	3	2	1	1	1

APPENDIX B

CONTROL DATA

This table displays the gypsum compatibility scores for the Control and Type IV dental gypsum (Prima Rock®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	2	2	2	2	2
3	1	1	1	1	1
4	2	2	2	1	1
5	1	1	1	1	1
6	2	2	2	2	2
7	1	1	1	1	1
8	2	2	2	2	2
9	1	1	1	1	1
10	1	1	1	1	1

APPENDIX C

CONTROL DATA

This table displays the gypsum compatibility scores for the Control and Type V dental gypsum (Die Keen®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	2	2	1	1	1
2	2	1	1	1	1
3	2	1	1	1	1
4	2	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	2	1	1	1	1



## APPENDIX D

### TEST DATA

This table displays the gypsum compatibility scores for Silgimix® and Type III dental gypsum (Microstone Golden®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1

## APPENDIX E

### TEST DATA

This table displays the gypsum compatibility scores for the Silgimix® and Type IV dental gypsum (Prima Rock®).

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1

APPENDIX F

TEST DATA

This table displays the gypsum compatibility scores for the Silgimix® and Type V dental gypsum (Die Keen®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1

APPENDIX G

TEST DATA

This table displays the gypsum compatibility scores for the Alginot® and Type III dental gypsum (Microstone Golden®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1

## APPENDIX H

### TEST DATA

This table displays the gypsum compatibility scores for the Alginot® and Type IV dental gypsum (Prima Rock®).

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1

APPENDIX I

TEST DATA

This table displays the gypsum compatibility scores for the Alginot® and Type V dental gypsum (Die Keen®).

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1

APPENDIX J

TEST DATA

This table displays the gypsum compatibility scores for the Position Penta® and Type III dental gypsum (Microstone Golden®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1

APPENDIX K

TEST DATA

This table displays the gypsum compatibility scores for the Position Penta® and Type IV dental gypsum (Prima Rock®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1



## APPENDIX L

### TEST DATA

This table displays the gypsum compatibility scores for the Position Penta® and Type V dental gypsum (Die Keen®)

Sample #	Immediate	24hrs	48hrs	168hrs	336hrs
1	1	1	1	1	1
2	1	1	1	1	1
3	1	1	1	1	1
4	1	1	1	1	1
5	1	1	1	1	1
6	1	1	1	1	1
7	1	1	1	1	1
8	1	1	1	1	1
9	1	1	1	1	1
10	1	1	1	1	1