

THE EFFECT OF TEMPERATURE ON THE LINEAR DIMENSIONAL STABILITY OF ELASTOMERS.

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Health Sciences and WHO Oral Health Collaborating Centre,

University of the Western Cape



Supervisor

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DECLARATION

I hereby declare that “The effect of temperature on linear dimensional stability of elastomers” is my own work, that it has not been submitted before for any degree or examination in this or any other university, and that all the sources I have used or quoted have been indicated and acknowledged as complete references.

Dr. Susan Sanette Muller

Signed:

This 2th day of August, 2012



DEDICATION

To my loving husband and best friend Anton Muller for the great sacrifice he made to take care of our lovely children while I was studying, for his never ending support and encouragement throughout the course and for believing in me.

To my children, Ruben, Simone and Zander for your unconditional love, you are my light.

To my parents, brother and sister, relatives, friends and colleagues who supported me through the course of this degree.



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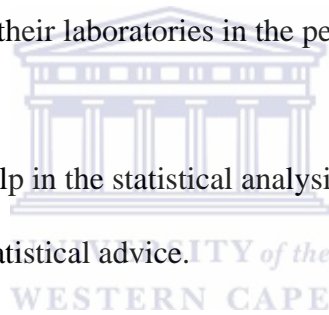
I wish to express my sincere gratitude to the following people for their unconditional assistance in developing this research project.

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ABSTRACT

Title: The effect of temperature on linear dimensional stability of elastomers.

Objectives: Sometimes, dental impressions need to be transported to distant laboratories. It has been reported that the temperature in a vehicle can reach up to 66°C when the outdoor temperature is 38°C. These temperatures may be reached during South African summers. The objective of this *in vitro* study was to investigate the effect of temperature and time on the dimensional stability of two elastomeric impression materials.

Methodology: Specimens consisted of impressions made of an ISO-specified test-block featuring a pattern of grooves. Materials used were polyether (Impregum Penta) and polyvinylsiloxane (Affinis Precious regular body). Using an SLR camera and standardized technique, the specimens were photographed at 2 different temperatures (21°C and 66°C) and 3 time intervals (30min, 8hrs and 14 days). This resulted in a total of 12 groups (n=10) to be compared. Digital images of the impressions were calibrated and measured using digital analyzing software. These distances were used to evaluate the mean % dimensional change (%DC) for each group. VEPAC module of Statistica 10 was used for the statistical analysis. To analyze exactly where the differences lied, a Fisher LSD correction was applied to correct for multiple pair wise comparisons.

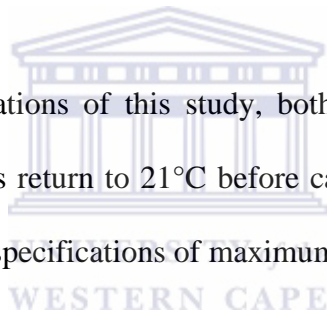
Results: Comparing polyether with silicone, there was no difference in the mean %DC for specimens kept at 21°C for 8hrs (polyether=0.364; silicone=0.237). Neither was there a difference between polyether and silicone when heated to 66°C, cooled off, and measured

after 8hrs (polyether=0.306; silicone=0.297) or after 14 days (polyether=-0.272; silicone=-0.093).

For both polyether and silicone, the mean %DC of the groups exposed to 66°C, cooled off and measured after 8hrs (polyether=0.306; silicone=0.297) differed significantly when compared to the group measured after 14 days (polyether=-0.2723; silicone=-0.092) ($P<0.0001$ and $P=0.0029$ resp).

For both polyether and silicone, the mean %DC of the groups exposed to 66°C, cooled off and measured after 8hrs (polyether=0.306; silicone=0.297) did not differ when compared to the 21°C (polyether=0.364; silicone=0.237).

Conclusions: Within limitations of this study, both materials were heat-sensitive. It is recommended that materials return to 21°C before casting. Despite statistical differences, all results were within ISO specifications of maximum 1.5%DC.



KEYWORDS

Impression materials

Elastomers

Linear dimensional stability

Temperature

Time

Digital image analysis



DEFINITIONS

For the purpose of this study the following terms will be defined based on the Glossary of Prosthodontics Terms (2005):

Elastomeric impression material: a group of flexible chemical polymers, which are either chemically or physically cross-linked. Generally, they can be easily stretched and rapidly recover their original dimensions when applied stresses are released.

Dimensional stability: the ability of a material to retain its size and form over time.

Impression material: any substance or combination of substances used for making an impression or negative reproduction.

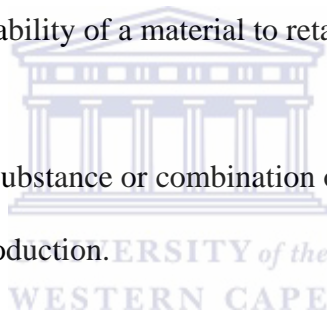


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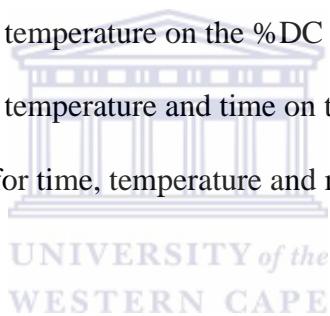


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CHAPTER 1.

INTRODUCTION AND LITERATURE REVIEW

1.1. INTRODUCTION

Impression materials are used in dentistry to make impressions of the teeth and surrounding structures. These impressions are used to manufacture diagnostic and master casts. Diagnostic casts are used to assist in treatment planning, while master casts are used to manufacture complete or partial removable prosthesis, fixed partial prosthesis and implant-supported prosthesis. These procedures cover most aspects of a prosthodontic practice, including intra-oral and extra-oral prostheses.

Even though new technology allows digital impression making, it is not expected that this technology will substitute traditional impression making in the foreseeable future.

Making impressions and casts is a complex procedure, requiring accuracy in the handling of a number of materials, such as tray materials, tray adhesives, impression materials, dental stones, as well as using appropriate clinical and laboratory techniques. The quality of the impression is crucial for the fit of the definitive prosthesis and contributes to the long-term success of the dental reconstruction. Information supplied by manufacturers should be tested and supported by independent research. Since dental materials are continuously modified in an effort to improve their clinical performance, independent research should also be ongoing.

1.2. LITERATURE REVIEW

1.2.1. IMPRESSION MATERIALS USED IN DENTISTRY

There are several types of elastomeric impression materials available for dental use. Table 1.1 (Rosentiel *et al.*, 2006) provides a list of the categories of impression materials for dental impression making. For work requiring a high level of fit, such as for fixed - and implant prosthodontics, the materials of choice are the polyvinyl siloxane and polyether impression materials, because they have a reputation for being accurate and dimensionally stable.

1.2.2. PROPERTIES OF IMPRESSION MATERIALS

Ideal properties

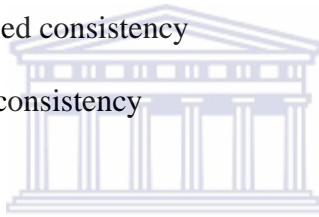


The search for an ideal impression material is ongoing. The properties of interest for impression materials include: detail reproduction, dimensional stability, elastic recovery, flexibility, flow, hydrophilicity, patient comfort, a long shelf life and economics (Donovan and Chee, 2004). Impression materials differ in relation to ideal properties, but these differences can provide a basis for selecting the most appropriate impression material for a specific clinical situation: one single impression material does not fit every clinical situation ideally. Table 1.1 (Rosentiel *et al.*, 2006) highlights the most relevant advantages, disadvantages and clinical applications of the different categories of impression materials.

Of course, the best possible material properties should be supported by the correct manipulation of the materials. Issues to be considered should be: adequate mixing, creating a uniform bulk, control over viscosity, tray selection, achieving adhesion to the tray and correct pouring of the impression (Donovan and Chee, 2004).

The International Standard Organization (ISO) classifies elastomeric impression materials according to consistencies determined directly after completion of mixing according to manufacturer's instructions (ISO 4823:2000):

- Type 0: putty consistency
- Type 1: heavy-bodied consistency
- Type 2: medium-bodied consistency
- Type 3: light-bodied consistency



According to ISO, type 0 (putty) and type 1 (heavy-bodied) materials produce an indent $\leq 35 \mu\text{m}$ when subjected to a consistency test. Impressions with these materials should be made in one or two steps. Type 2 (medium-bodied) materials produce an indent between 31 to 41 μm , and impressions are made in one step. Type 3 (light-bodied) materials produce an indent of $\geq 36 \mu\text{m}$, and a syringe is used for making the impression (ISO 4823, 2000). The viscosity of the setting material influences the accuracy or detail reproduction of an impression (Hamalian *et al*, 2011).

Table 1.1: Elastic impression materials (Adapted in part from Rosentiel et al., 2006).

Materials	Advantages	Disadvantages	Recommended use	Precautions
Reversible hydrocolloid	Hydrophilic Long working time Low cost	Low tear resistance Low stability Special equipment needed Advanced preparation required	Multiple preparations Problem with moisture	Pour immediately Use stone only
Irreversible hydrocolloid	Rapid set Easy manipulation Patient acceptance Low cost	Pour once with reasonable accuracy Limited accuracy and surface detail reproduction	Diagnostic casts Not for working casts.	Pour immediately.
Polysulphide polymer	High tear strength Comparatively easier to pour than other elastomers Accurate through third pour if poured immediately	Unpleasant odour Long setting time Stability not so fair Distortion over time Hydrophobic	Most impressions	Pour within 1 hour. Allow to set for 10 minutes
Polyether	Short setting time Auto-mix available Excellent dimensional stability and accuracy Acceptable for implant impressions	Set material rigid, Imbibition Short working time	Most impressions	Care not to break teeth when separating from cast.
Addition-cured silicone	Short setting time Auto-mix available Pleasant to use Excellent dimensional stability	Hydrophobic Poor wetting Some materials release H ₂	Most impressions	Delayed pour of some materials Care to avoid bubbles when pouring
Condensation-cured silicone	Short setting time Easy to use	Hydrophobic Poor wetting Low dimensional stability	Most impressions	Pour immediately Care not to incorporate bubbles

Two important properties of impression materials that contribute to an accurate reproduction of teeth and surrounding tissues are detail reproduction and dimensional stability. These properties will be discussed in the next two paragraphs.

Accuracy

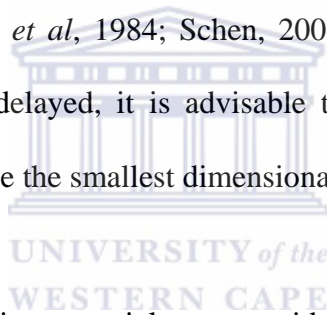
The accuracy of an impression material has 2 aspects: detail reproduction and dimensional accuracy. Although no definition for detail reproduction was found, the American Dental Association specification #19 prescribes that elastomeric impression materials should produce fine detail of 25 μ m or less. The ISO requires a continuous line width reproduction of 75, 50, and 20 μ m depending on the viscosity of the material. As mentioned earlier, the viscosity of the setting material influences the accuracy or detail reproduction of an impression (Hamalian *et al*, 2011). Derrien and Le Menn (1995 cited in Pant *et al.*, 2008) mentioned that polyvinyl siloxane impression materials could reproduce detail of 1 to 2 μ m. However, if conventional impressions and casts are used for fabricating indirect restorations, this level of accuracy of impression materials is largely lost because dental stone cannot reproduce detail finer than 20 μ m. The dimension of gypsum crystals ranges from 15-25 μ m. Detail reproduction of polyvinyl siloxane impression materials is time and product dependent (Pant *et al.*, 2008).

Dimensional stability

Dimensional stability differs from dimensional accuracy in the sense that

dimensional stability measures change over time. The Glossary of Prosthodontic Terms (2005) defines dimensional stability as the ability of a material to retain its size and form over time. An ideal impression material should be dimensionally stable reflecting its property to be dimensionally accurate at any given time after impression making, allowing the operator to pour it at any convenient time.

Because of its time-dependency the greatest dimensional accuracy occurs immediately after polymerization (Rubel, 2007). Accuracy is lost with extended periods of time (Schen, 2003; Donovan and Chee, 2004), because of lack of dimensional stability. Polyether and polyvinyl siloxane are accurate for 1 – 2 weeks, whereas polysulfide is accurate if the impression is casted within between 1 - 2 hours (Williams *et al*, 1984; Schen, 2003) of impression making. Therefore, should pouring be delayed, it is advisable to use addition-silicone or polyether materials, which have the smallest dimensional change over time.



Elastomeric impression materials are considered to be more dimensionally stable than hydrocolloid impression materials. Hydrocolloid materials should be poured within 10 minutes after removal from the mouth. These materials are composed of 80% water and are subject to syneresis (evaporation of water) and imbibition (absorption of water), resulting in distortion. Therefore it is important to avoid wrapping these impressions in a wet paper towel, as seems to be common practice. This may result in absorption of water from the paper towel and cause distortion of the impression (Donovan and Chee, 2004).

Even though they are considered to be superior to hydrocolloids in terms of dimensional stability, elastomers are subject to small changes. All currently available elastomeric impression materials undergo polymerization shrinkage (Rubel, 2007). This is caused by the rearrangement of the bonds during

polymerization. Additional shrinkage may occur when unstable by-products, formed during polymerization, evaporate. Therefore, polysulfide impression materials and condensation silicones have the largest dimensional change during setting. Polyethers exhibit a slight change, whereas addition silicones have the smallest change. The shrinkage for these two products is lower because there is no loss of by-products. (Williams *et al.*, 1984; Anusavice, 2003; Donovan and Chee, 2004)

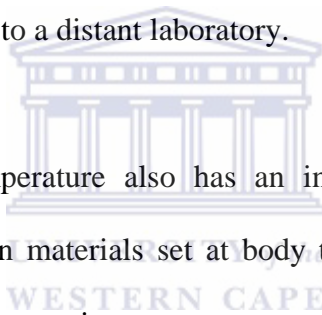
Addition-silicones acquire almost the ideal dimensional stability because there are no by-products formed during the chemical setting reaction. It can be poured immediately after the impression has been removed from the oral cavity, or days after impression making and can still produce an accurate cast if poured within 1 – 2 weeks (Donovan and Chee, 2004). Marcinak and Draughn (1982) investigated delayed pour for up to 1 week and found no significant change (-0.3%) with addition-cured silicones. Williams *et al.* (1984) and Johnson and Craig (1986) agreed and reported that addition-cured silicones were the most accurate after delayed pour of elastomeric impression materials.

Chen *et al.* (2004) investigated storage time and filler loading of elastomers and found the greatest accuracy on polyvinyl siloxane and highly-filled materials.

Condensation silicones produce ethyl alcohol, and polysulfide rubber impression materials produce water as a by-product as a result of the setting reaction (Donovan and Chee, 2004). These by-products can evaporate from the surface of the set impression. Over time these materials shrink. Therefore impressions made of these materials should be poured in less than 30 minutes after removal from the mouth to prevent distortion. In 1981, Lacy *et al.* investigated delayed pour of elastomers and

concluded condensation silicone systems should be poured as soon as possible after making the impression.

Polyether impression materials are subject to imbibition. This material swells over time due to water absorption from the atmosphere, resulting in a distorted impression (Lacy *et al.*, 1981; Williams *et al.*, 1984). It is recommended that for optimal accuracy polyether impression materials should be poured within 60 minutes after removal from the mouth (Donovan and Chee, 2004). Due to its property of absorbing water from the atmosphere, care should be taken not to store polyether impression material together with water-based impression materials when sending impressions to a distant laboratory.



It appears that temperature also has an influence on accuracy of impression materials. Impression materials set at body temperature. However, after removal from the mouth, they acquire room temperature. De Araujo and Jorgensen, (1986) found improved accuracy by reheating addition-reaction silicone impression, from room temperature to 37°C. If even these relatively small temperature changes have an effect on accuracy, what would the effect be of larger temperature fluctuations? Purk *et al.*, (1998) investigated elastomers under extreme temperature and storage time. He found that all elastomers were unstable under extreme temperatures.

According to Purk *et al.* (1998), it is not unusual for parcels to stay in delivery vehicles for more than eight hours during the delivery process. It has been reported that the temperature in a delivery vehicle can reach up to 66°C when the outdoor temperature is 38°C. During South African summers, these outside temperatures can be reached.

Corso *et al.*, (1998) tested the effect of temperature changes on the dimensional stability of polyvinyl siloxane and polyether impression materials. They found that changes in storage temperature had a statistically significant effect on the dimensional stability of horizontal and vertical lines. Overall dimensional changes were very small. These measurements were made on a master cast poured from the impression and not on the impression itself.

Pant *et al.*, (2008) investigated delayed pour and different storage temperatures of polyvinyl siloxane duplicating impression materials and found the dimensional change to be less than 2%.

1.2.3. TESTING OF ELASTOMERIC IMPRESSION MATERIALS



Many scientific studies examined the influence of different variables, such as time, storage conditions and clinical technique, during impression making and casting of the impressions. However, these studies often introduced more than one possible variable and the true dimensional stability or accuracy of the impression material itself is not established, rather the accuracy of the procedure as a whole is assessed (Piwowarczyk *et al.*, 2002).

Methods

There is no consistency in the literature in terms of methodology used for assessing dimensional stability of impression materials. Some publications report the use of a standard, but most publications describe some kind of clinically related technique incorporating unknown variables such as impression tray and casting materials.

Measurements are made of the cast or of the impression itself. This lack of standardization makes direct comparisons among different studies difficult.

Standards

Several standards are developed for testing impression materials.

The American Dental Association (ADA) specification No. 19 introduced a standardized repeatable method in 1977 for preparing and evaluating test specimens of elastomeric material.

The British Standards Institution (BSI) employs a scribed block, which is used to form a disc of impression materials (British Standards Institution, 1987).

The International Organization for Standardization (ISO) is a worldwide federation of national standards bodies (ISO member bodies). They published a standard for testing and minimum requirements for elastomeric impression materials for dentistry (ISO 4823:2000). This standard describes in detail the manufacturing of the specimens using a custom-made test block with a special configuration of lines for determining detail reproduction and dimensional stability. The measurements are done on the impression material itself, eliminating any variable introduced by tray or casting procedures and materials.

There seems to be no consensus in the literature on the measuring device that should be used to evaluate the detail reproduction and dimensional stability of impression materials. Microscopes, travelling microscopes, calipers and scanners are used. Manual measuring devices are easy to use, however error due to operator fatigue must be accepted. It is also important to note that these standards date back

to 1977, 1987 and 2000 and more modern measuring techniques are available now.

1.3. CONCLUSION

Impression making is a fundamental part of prosthodontic practice. A literature review indicated that few studies report on the influence of prolonged raised temperatures, as may be found during transport, on dimensional stability. Therefore the aim of this study is to investigate the effect of raised temperatures on the linear dimensional stability of elastomeric impression material.



CHAPTER 2: AIMS AND OBJECTIVES

2.1. AIM

The aim of this *in vitro* study was to investigate the influence of temperature and time on the linear dimensional stability of a polyether and a silicone impression material.

2.2. OBJECTIVES

The objectives of this study were:

- a. To establish if an increase in *temperature* influences the linear dimensional stability of a polyether impression material.
- b. To establish if an increase in *temperature* influences the linear dimensional stability of a polyvinyl siloxane impression material.
- c. To establish if *time* influences the linear dimensional stability of a polyether impression material.
- d. To establish if *time* influences the linear dimensional stability of a polyvinyl siloxane impression material.

2.3. NULL HYPOTHESES

The null-hypotheses to be tested were:

- a. *Temperature* has no influence on the linear dimensional stability of polyether or polyvinyl siloxane impression materials.
- b. *Time* has no influence on the linear dimensional stability of polyether or polyvinyl siloxane impression materials.

CHAPTER 3: MATERIALS AND METHODS

3.1. INTRODUCTION

This *in vitro* controlled comparative study assessed the influence of time and temperature on the linear dimensional stability of 2 different elastomeric impression materials.

The proposal was approved by the research and ethics committee of the University of the Western Cape. The study was conducted in the Department of Restorative Dentistry, Tygerberg Oral Health Center at the University of the Western Cape.

The fabrication of the specimens was done according to the specifications of the International Organization of Standardization (ISO) 4823:2000 for testing dental elastomeric impression materials. The measuring of the specimens was done digitally and not by means of a travelling microscope as is specified by ISO.

3.2. METHODOLOGY

3.2.1. Stainless steel test block

A stainless steel test block and ring was manufactured according to the specifications of ISO 4823:2000 for testing dental elastomeric impression materials. The surface of the test block was marked with 3 horizontal lines (no. 1, 2 and 3) intersected by 2 vertical lines (no. 4 and 5) (Fig 3.1).

Impressions of the test block's surface were made. The distance between lines 4 and 5 on the impressions' surface was digitally measured 3 times. (Figure 3.1)

Only impressions that passed the ISO requirements for detail reproduction were used for dimensional stability measurements: a line reproduction was considered satisfactory if the required line 1,2 or 3 is continuous between the lines 4 and 5 (See test block in Figure 3.1). Impressions that failed the ISO requirements for detail reproduction were not used for the dimensional stability measurements.

Figure 3.1: Measuring the distance between line no. 4 and 5 along line 3 (ISO 4823).



The average of the 3 measurements was used to calculate the percentage of dimensional change for each specimen to the nearest 0,000000001% using the equation:

$$\% \text{ DC} = 100 [L1 - L2 / L1]$$

with L1= the distance measured on the test block and L2 = the distance measured on the specimen. According to ISO requirements, a specimen passes the dimensional stability test if the DC is not more than 1.5%.

3.2.2. Impression Materials

The impression materials used were a polyvinyl siloxane material (S) (Affinis® Precious regular body, Coltene/whaledent®) and a polyether material (P) (Impregum™ Penta™, 3M ESPE). Both are type 2 elastomeric impression materials. See addendum 1 for material specifications.

The impression materials were handled according to manufacturers' instructions.

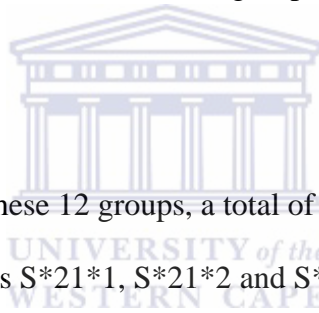
3.2.3. Groups

Twelve groups of 10 specimens each were developed. The treatment of the specimens in the 12 groups was as follows:

- Group S*21*1 (control): silicone impression stored at room temperature ($21^{\circ}\text{C}\pm 1$), photographed after 30 minutes.
- Group S*21*2: silicone impression stored at room temperature, photographed after 8 hrs.
- Group S*21*3: silicone impression stored at room temperature, photographed after 14 days.
- Group S*66*1: silicone impression stored at 66°C for 8 hrs before photographed.
- Group S*66*2: silicone impression stored at 66°C for 8 hrs, cooled down to room temperature before photographed.
- Group S*66*3: silicone impression stored at 66°C for 8 hrs and photographed at room temperature after 14 days.
- Group P*21*1 (control): polyether impression stored at room temperature, photographed after 30 minutes.

- Group P*21*2: polyether impression stored at room temperature, photographed after 8 hrs.
- Group P*21*3: polyether impression stored at room temperature, photographed after 14 days.
- Group P*66*1: polyether impression stored at 66°C for 8 hrs before photographed.
- Group P*66*2: polyether impression stored at 66°C for 8 hrs, cooled down to room temperature before photographed.
- Group P*66*3: silicone impression stored at 66°C for 8 hrs and photographed at room temperature after 14 days.

A summary of the identification of the groups and their treatment is given in Table 3.1.



In order to develop these 12 groups, a total of 40 specimens had to be made.

Specimens for groups S*21*1, S*21*2 and S*21*3 were the same (n=10)

Specimens for groups S*66*1, S*66*2 and S*66*3 were the same (n=10)

Specimens for groups P*21*1, P*21*2 and P*21*3 were the same (n=10)

Specimens for groups P*66*1, P*66*2 and P*66*3 were the same (n=10).

3.2.4. Specimen fabrication

Before use, the test block and ring were ultrasonically cleaned and placed in an oven, set at $35 \pm 1^\circ\text{C}$ for at least 15 min for conditioning. All the impressions were made using prepackaged cartridges of polyether and polyvinyl siloxane with a micro plastic dispenser MKII (Coltene/whaledent®) and the Pentamix 2 electric mixing unit (3M ESPE) respectively. The ring was positioned over the block and the cavity that is formed in this way was filled with impression material. A

polyethylene-covered glass plate was placed over the ring and pressed down until excess material was expelled. A thin film of silicone grease was sprayed over the plate to help secure the polyethylene sheet to the plate. At 60s after completion of the mix, this specimen-forming assembly was placed in a water bath at 37°C for the minimum time recommended by the manufacturer’s instructions for leaving the impression in the mouth (Affinis: 3:00 min & Impregum: 3:15 min). Consequently, the impression material specimen in the ring mould was separated from the test block. The specimen surface was flushed with distilled water and dried using a gentle stream of clean air. The specimens were treated according to the regimens indicated for the 12 groups (Table 3.1).

Table 3.1: This table shows for each group the *exposure to temperature, time interval between impression taking and reading and conditions* at which readings are made. P = polyether, S = silicone.

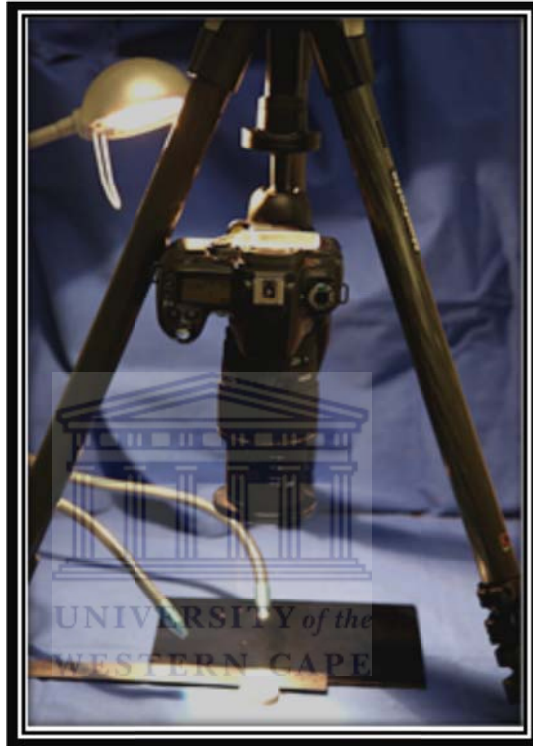
Group	Temperature (°C)	Time	Conditions
S*21*1 & P*21*1	21	30min	Read @ 21°
S*21*2 & P*21*2	21	8hrs	Read @ 21°
S*21*3 & P*21*3	21	14days	Read @ 21°
S*66*1 & P*66*1	66	8hrs	Read @ 66°
S*66*2 & P*66*2	66	8hrs	-----
	21	8hrs	Read @ 21°
S*66*3 & P*66*3	66	8hrs	-----
	21	14days	Read @ 21°

3.2.5. Digital Image Analysis

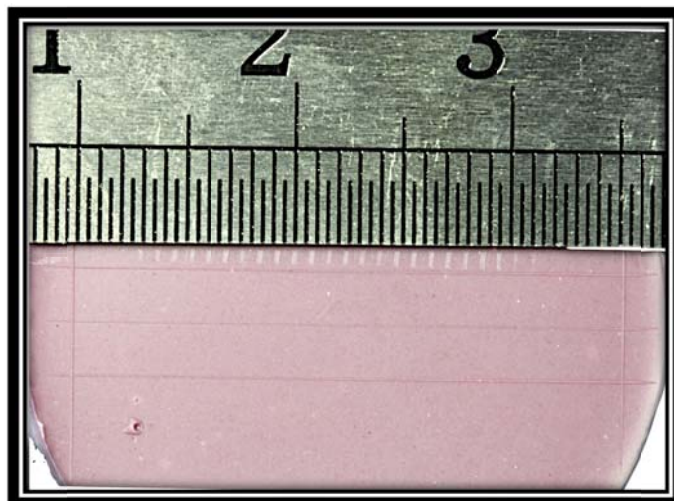
Images of the specimens were taken at the time/temperature conditions as specified

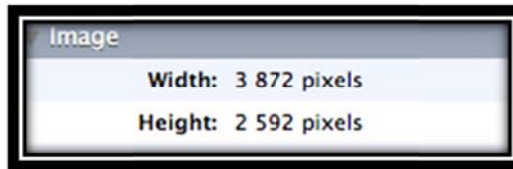
in the last two columns of table 3.1. The specimens, together with a ruler (0.5 mm scale) were photographed with a SLR digital camera (Nikon D80, macro lens) using a standardized technique: sensor, specimen and ruler were all mounted parallel to each other (Figures 3.2 and 3.3)

Figure 3.2: Mounting of the camera and specimen for the digital imaging.



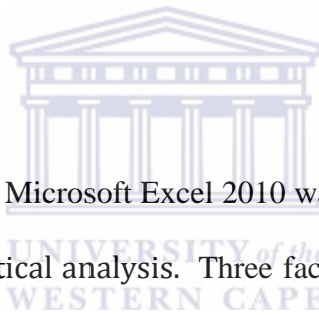
*Figure 3.3: Image of a specimen with calibrating ruler.
The dimension of the images was 3872 x 2592 pixels.*





The software used for measuring the images was AnalyzingDigitalImages, version 11, August 2008. This software was developed by the Museum of Science, Boston, and may be freely downloaded and used for educational purposes (<http://mvh.sr.unh.edu>). It allows scaling, calibration and analysis of digital images up to a magnification of 9. The distances were measured up to 0.00001mm accuracy (0.01 μ m).

3.3. DATA ANALYSIS



For the descriptive statistics Microsoft Excel 2010 was used. VEPAC module of Statistica 10 was used for the statistical analysis. Three factors that were considered: a) material type (P or S); b) temperature (21 or 66); and time point for measurement (1, 2 or 3). It was this factor that caused the repeated measurement of the same sample, resulting in repeated measures analysis of variance (ANOVA). Interactions between all variables were also analyzed. To analyze exactly where the differences lay, a Fisher LSD correction was applied to correct for multiple pairwise comparisons. The normal probability plot of the mixed model ANOVA was checked for normality and judged to be not a problem (results not shown).

Twenty comparisons were selected from the multiple pairwise comparisons (addendum 5) to establish the effect of time and temperature on the dimensional stability of the 2 impression materials:

To establish the effect of:

1. *different materials* on the DS, the following groups were compared:

S*21*1	&	P*21*1
S*21*2	&	P*21*2
S*21*3	&	P*21*3
S*66*1	&	P*66*1
S*66*2	&	P*66*2
S*66*3	&	P*66*3

2. *time* on the DS the following groups were compared:

S*21*1	&	S*21*2
S*21*2	&	S*21*3
S*21*1	&	S*21*3
S*66*2	&	S*66*3
P*21*1	&	P*21*2
P*21*2	&	P*21*3
P*21*1	&	P*21*3
P*66*2	&	P*66*3

3. *exposure to temperature* on the DS the following groups were compared:

S*21*2	&	S*66*2
S*21*3	&	S*66*3
P*21*2	&	P*66*2
P*21*3	&	P*66*3

4. *temperature of the specimen at the time of reading* on the DS the following groups were compared:

S*66*1	&	S*66*2
P*66*1	&	P*66*2

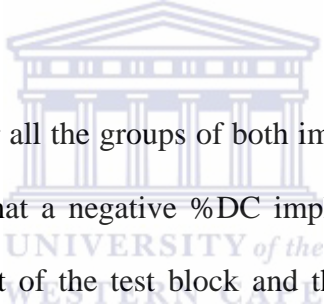
CHAPTER 4: RESULTS

4.1. INTRODUCTION

All specimens passed the ISO requirements for detail reproduction. Therefore, no specimens had to be replaced.

The complete data set consisting of the raw data, percentage dimensional change (%DC) for all groups and multiple comparisons are shown in Addendum 4, 5 and 6.

4.2. DESCRIPTIVE STATISTICS



The descriptive statistics for all the groups of both impression materials are represented in Tables 4.1 and 4.2. Note that a negative %DC implies that the linear dimension of the specimen is larger than that of the test block and that a positive %DC implies that the specimen has linear dimension smaller than that of the test block. For the determination of the minimum %DC, the value closest to zero %DC is chosen and for the maximum %DC the value furthest away from zero %DC is chosen.

Table 4.1: Summary of the descriptive statistics (%DC) for the polyether impression material (P).
(St Dev = standard deviation)

Group	P*21*1	P*21*2	P*21*3	P*66*1	P*66*2	P*66*3
Mean	0.120188	0.364424	-0.038932	0.202031	0.306417	-0.272346
Median	0,114914	0.389232	0.039426	0.229160	0.313170	0.259654
Minimum	0.014910	0.230039	0.004073	0.084989	0.206713	-0.092288
Maximum	0.237458	0.432349	-0.217358	0.607219	0.417062	-0.447477
St Dev	0.059507	0.064329	0.091504	0.240434	0.072987	0.121282

The lowest mean %DC for the polyether material was found for the group that was left for 2 weeks at room temperature before measurements were made (group P*21*3 with a mean %DC=-0.038932). The highest mean %DC was found for group that was kept at 21°C for 8 hours (P*21*2, %DC = 0.364424).

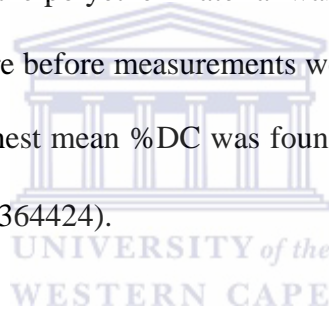


Table 4.2: Summary of the descriptive statistics (%DC) for the silicone impression material (S).
(St Dev = standard deviation)

Group	S*21*1	S*21*2	S*21*3	S*66*1	S*66*2	S*66*3
Mean	-0.086977	0.237352	0.005887	-0.094716	0.297070	-0.092751
Median	0.115694	0.237160	0.026117	0.098331	0.273043	0.084731
Minimum	0.037846	0.181363	0.011346	0.053560	0.190540	-0.036775
Maximum	-0.215824	0.300456	-0.094338	-0.151028	0.426583	-0.167174
St Dev	0.101006	0.036468	0.051674	0.058153	0.085290	0.043492

The lowest mean %DC for the silicone impression material was found for the group that was left for 2 weeks at room temperature before measurements were made (group S*21*3 and %DC 0.005887). This was the group with %DC lower than the lowest %DC of the p-material. The highest mean %DC was found for group that was heated to 66°C for 8 hrs and cooled off to room temperature for another 8hrs (group S*66*2 with a %DC of 0.29707).

4.3 STATISTICAL ANALYSIS

Results from the fixed effect test for %DC indicated the presence of statistically significant differences (Table 4.3)

Table 4.3: Fixed effect test for %DC

Effect	Num. DF	Den. DF	F	p	
Material	1	36	11.54	0.00	<0.01
Temperature	1	36	4.38	0.04	
Time	2	72	188.34	0.00	<0.01
Material*Temperature	1	36	1.77	0.19	
Material*Time	2	72	37.52	0.00	<0.01
Temperature*Time	2	72	13.27	0.00	<0.01
Material*Temperature*Time	2	72	4.41	0.02	

Figure 4.4: Effect of different materials on the %DC.

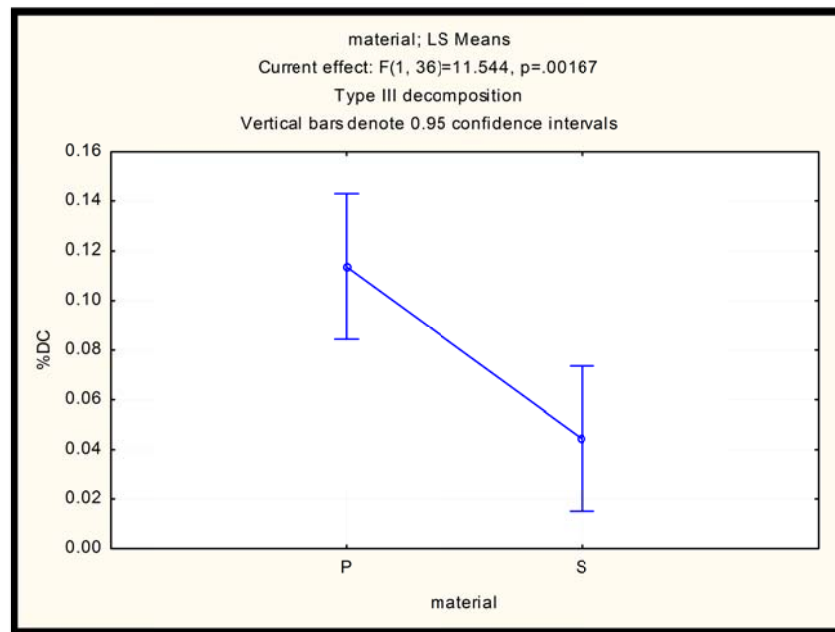


Figure 4.4 demonstrates the effect of different *materials* on the %DC. To establish the effect of *material* on linear dimensional stability, pairs of groups subjected to the same time/temperature regimen for each material were compared with each other (Table 4.4). There was a significant difference in %DC for all the pairs, except between the groups left for 2 weeks at room temperature (S*21*3 & P*21*3) and between the groups exposed to 66°C, cooled off and measured after 8hrs (S*66*2 & P*66*2).

Table 4.4: This table shows pairs of groups and p-values to demonstrate the effect of different materials on the %DC. Red p-value represents significant difference.

Groups		P-value
S*21*1	P*21*1	0.000016
S*21*2	P*21*2	0.0059650
S*21*3	P*21*3	0.320918
S*66*1	P*66*1	0.000000
S*66*2	P*66*2	0.835480
S*66*3	P*66*3	0.000149

Figure 4.5: The effect of time on the mean %DC. 1 = after 30 min of making the impression; 2 = after 8hrs; 3= after 14 days.

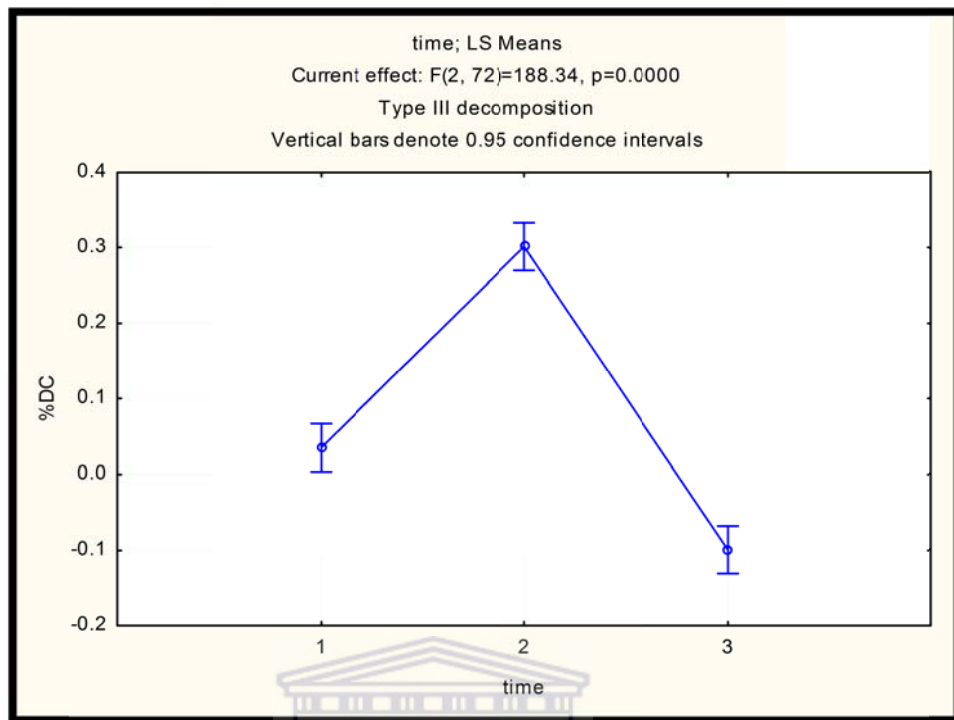


Figure 4.5 demonstrates the effect of *time* on the %DC. To establish the effect of *time* on the linear dimensional stability the groups in table 4.5 were compared. All pairs showed a significant difference in %DC.

Table 4.5:

This table shows the pairs of groups and *p*-values to demonstrate the effect of *time* on the %DC. Red *p*-values represent significant differences.

Groups		P-value
S*21*1	S*21*2	0.000000
S*21*2	S*21*3	0.000001
S*21*1	S*21*3	0.030365
S*66*2	S*66*3	0.000000
P*21*1	P*21*2	0.000000
P*21*2	P*21*3	0.000000
P*21*1	P*21*3	0.000316
P*66*2	P*66*3	0.000000

Figure 4.6: The effect of temperature on the %DC.

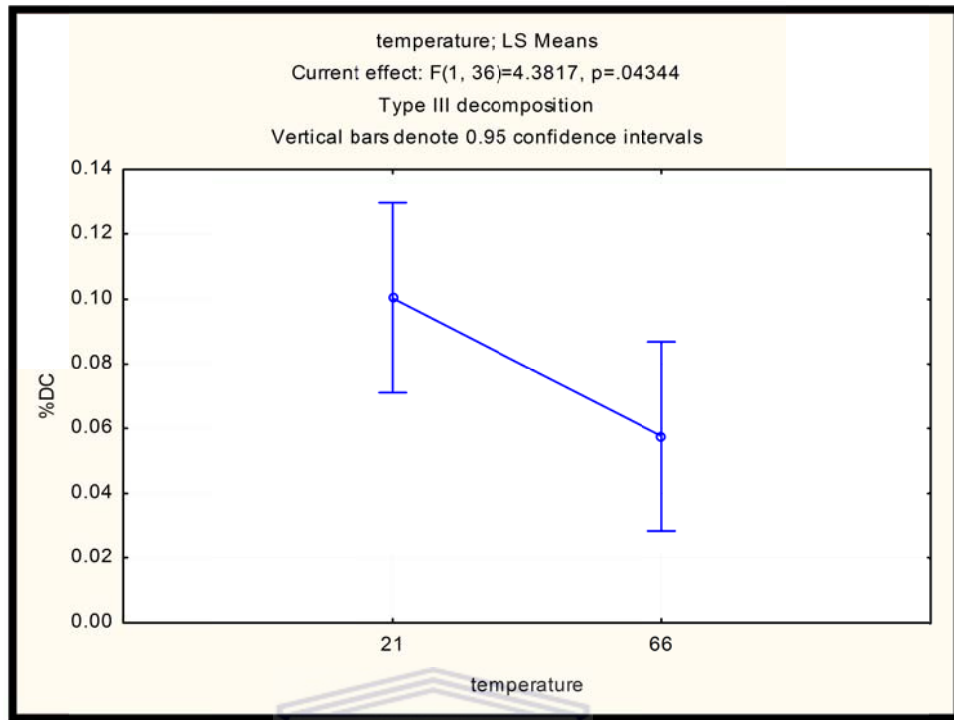


Figure 4.6 demonstrates the effect of only *temperature* on the %DC. To establish the effect of *exposure to temperature* on dimensional stability, the groups in table 4.6 were compared.

Table 4.6

This table shows the pairs of groups and p-values to demonstrate the effect of exposure to temperature on the %DC.

Red p-values represent significant differences.

Groups		P-value
S*21*2	S*66*2	0.187143
S*21*3	S*66*3	0.031040
P*21*2	P*66*2	0.199941
P*21*3	P*66*3	0.000002

For silicone, the specimens that were kept at room temperature for 14 days (S*21*3) and the specimens that were exposed to 66°C for 8hrs and then left at room temperature for 14

days (S*66*3), differed significantly with a p-value of 0.031040.

Likewise, for polyether, the specimens that were kept at room temperature for 14 days (P*21*3) and the specimens that were exposed to 66°C for 8hrs and then kept at room temperature for a further 14 days (P*66*3), differed significantly with a p-value of 0.000002.

Figure 4.7: The effect of temperature and time on the mean %DC.

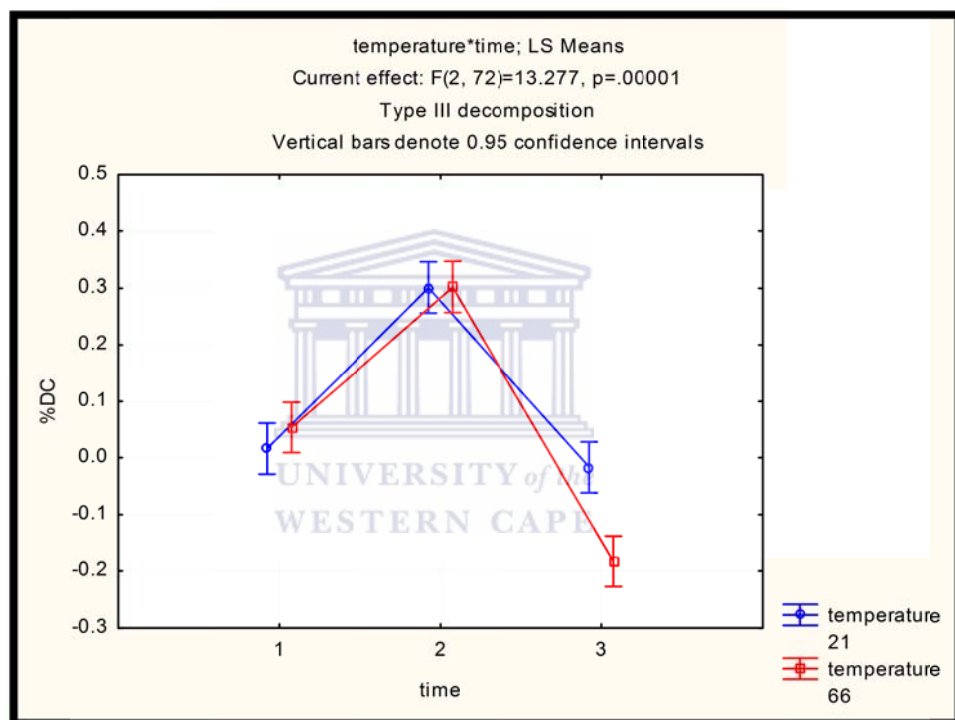


Figure 4.7 demonstrates the effect of *temperature and time* on the %DC. As can be seen from table 4.7, cooling off from 66°C to room temperature (S*66*1 & S*66*2) caused a difference in %DC (p=0.000000) for the silicone impression material. There was also a significant difference when polyether was cooling off from 66°C to room temperature (P*66*1 & P*66*2) with a p-value of 0.015362.

Table 4.7

This table shows the pairs of groups and p-values to demonstrate the effect of temperature of the specimens at the time of reading on the %DC. Shaded p-values represent significant differences

Groups		P-value
S*66*1	S*66*2	0.000000
P*66*1	P*66*2	0.015362

Figure 4.8 summarizes the effects of all the variables on the mean %DC. It is important to keep in mind that times 1, 2 and 3 for the blue and the red line are not the same (see also table 3.1). For the blue line, time 1 = 30 minutes and for the red line it is 8 hours. Time 2 for the blue line is 8 hours and for the red line it is 16 hrs (8 hrs at 66°C and an additional 8hrs for returning to 21°C). The same letters indicate no statistical difference between groups, whereas different letters indicate statistical difference between groups.

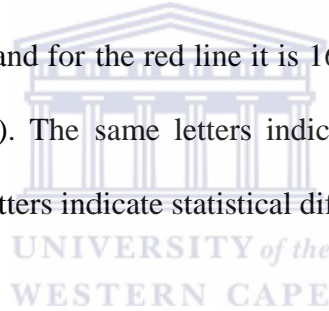
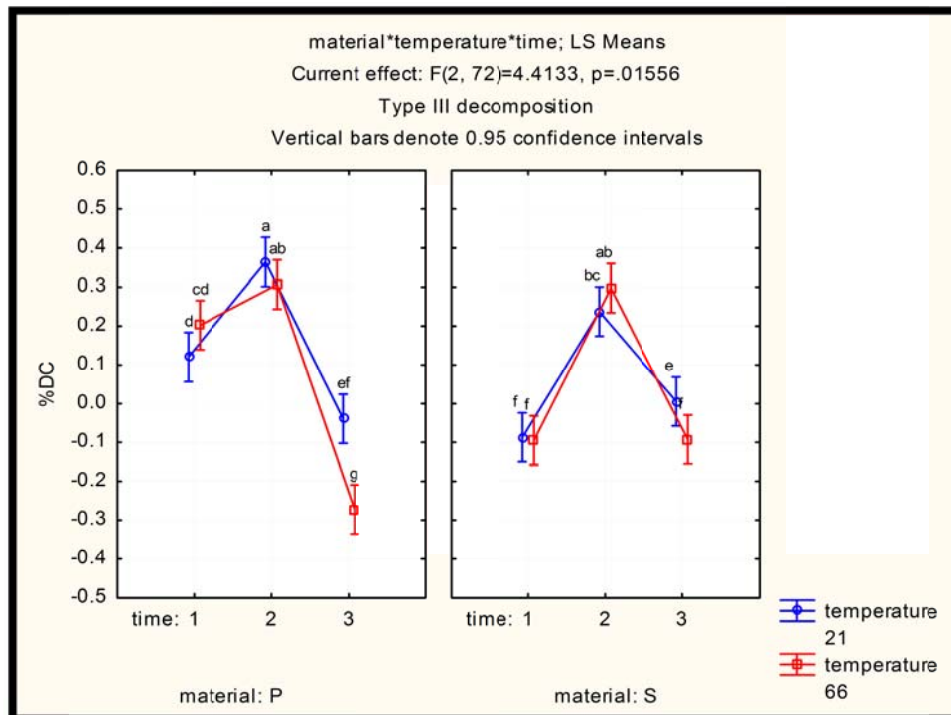


Figure 4.8. Mean %DC for time, temperature and materials



CHAPTER 5: DISCUSSION AND CONCLUSIONS

5.1 DISCUSSION OF THE RESULTS

This study investigated the influence of temperature and time on the linear dimensional stability of 2 types of elastomeric impression materials. The null-hypotheses stating that *temperature* or *time* have no influence on the linear dimensional stability of elastomeric impression materials, is rejected.

Even though statistical differences were found, it is important to remember that all the groups for both materials showed a mean dimensional change (DC) considerably lower than the recommended DC of not more than 1.5%.

For the explanation of the statistical results in the following discussion, it is helpful to refer to figure 4.8.

The linear dimensional stability of both materials is influenced by temperature, albeit in different ways.

Given enough time to recover (14 days at room temperature) after an 8hr period of exposure to an elevated temperature, the silicone impression material returns to its original dimension. This is evidenced by no statistical difference between groups S*66*1 & S*66*3 or S*21*1 & S*66*1. However, keeping the silicone impression material at room temperature for 14 days resulted in the highest dimensional accuracy. This accuracy was even higher than at 30 minutes after impression making. This difference was significant.

However, for polyether impression material a 14 day recovery at room temperature (after an extended period of exposure to an elevated temperature) resulted in an expansion. This expansion was significant.

For silicone specimens, the group that was left for 2 weeks at room temperature (S*21*3) was compared with the group that was exposed to 66°C for 8hrs and then left at room temperature for another 2 weeks (S*66*3), there was a significant difference with a p-value of 0.031040.

For polyether, the group that was left for 14 days at room temperature (P*21*3) was compared with the group that was exposed to 66°C for 8hrs and then left at room temperature for another 2 weeks (P*66*3), there was a significant difference with a p-value of 0.000002. The largest difference in %DC happened between the above-mentioned groups. Lacy *et al.* (1981) and Williams *et al.* (1984) reported that polyether material seem to be affected by imbibition resulting in expansion. The results of my research seem to confirm this. In addition, the group exposed to 66°C showed more expansion than the 21°C group. However, this effect was only apparent at 14 days. At 30 minutes and 8 hrs the linear dimensions were actually smaller. The smaller dimension may be due to polymerization shrinkage (Rubel, 2007). Modern materials still seem to be affected by dimensional fluctuations attributed to polymerization shrinkage and imbibition as previously described by Lacy *et al.* (1981), Williams *et al.* (1984) and Rubel (2007).

Exposing impression materials to an elevated temperature introduces another variable: *time*. Interpreting the results, these 2 variables must be analyzed together. For that reason a group of specimens not exposed to a higher temperature, but exposed to the same timeline acted as a control. For example, polyether that was kept at room temperature for 14 days

(P*21*3), was closer to 0 %DC than any other time or temperature group. Polyether that was heated to 66°C and allowed to cool off and recover for 14 days (P*66*3), expanded significantly. Therefore, it is concluded that it is the temperature and not the time that caused the difference.

To establish the effect of *material* on dimensional stability, pairs of groups subjected to the same time/temperature regimen for each material were compared with each other (Table 4.4). The 2 materials behaved differently for all time/temperature scenarios, except for 2 pairwise comparisons: Firstly, when left for 2 weeks at room temperature, there was no significant difference in %DC between the two materials. The mean %DC was lower for the silicone material compared to the polyether. And secondly, there was also no difference between the two materials measured after 8 hours when previously exposed to 66°C. This may be due to the polyether material absorbing moisture during storage time, as previously suggested by Lacy *et al* (1981) and Williams *et al.* (1984). Modern materials still seem to be affected by the imbibition described by these authors.

When specimens were kept at room temperature, there was a significant difference between the %DC when measured at 30 minutes and 8 hours after impression making, and also between the %DC measured at 8 hours and 14 days. There was also a significant difference between the 30 minute and 14 days readings, except for the silicone group that was not heated (Table 4.4). This phenomenon compensated for the initial expansion of the material in the first 30 minutes, and resulted in the most dimensionally accurate group of the complete study (S*21*3). After 8 hrs, for both materials and temperatures, the %DC was the highest (i.e. S*21*2; S*66*2; P*21*2; P*66*2 were the most dimensionally inaccurate).

Exposing both materials to 66°C for 8 hours compared to keeping them at 21°C did not have a significant effect on the %DC (Table 4.6).

However, the temperature of the specimens at the time of recording had a significant effect on the %DC for silicone, but not for the polyether. This was previously described by De Araujo and Jorgensen (1986), who recommended that impressions should be warmed to body temperature before pouring. Therefore, it is recommended that, in hot climates, silicone impressions are allowed to return to room temperature when they are delivered to the laboratory.

To investigate the influence of temperature, the specimens were subjected to a temperature for 66°C for the duration of 8 hours. This temperature was chosen because it was recorded as a temperature that occurred in delivery vehicles during American summers when the outside temperature was 32°C. During South African summers, these temperatures are easily reached. The time interval of 8 hours was an arbitrary time period for both the 21°C and 66°C groups of specimens to allow comparison between the 2 groups and it was thought that 8 hours might be a realistic time lapse between impression making in the surgery and fabrication of the cast in the laboratory.

Piwowarczyk *et al.* (2002) reported that detail reproduction and dimensional stability of impression materials can only be determined when any other variable is excluded from the methodology. Following the ISO methods, in this study the specimens themselves were measured, and not reproductions by means of casts. Also, no support by means of impression tray material was necessary. This eliminated any variable introduced by casting procedures and additional materials used in the process such as dental stones, tray and adhesive materials. Therefore, dimensional changes in this study are exclusively attributed to the impression material. Unfortunately, most scientific studies investigating dimensional

stability of elastomeric impression materials have included these variables, obscuring the real property of the material and making comparison among studies difficult. The accuracy of elastomeric impression materials is higher than that of gypsum casts (Donovan and Chee, 2004). Therefore, measuring casts in order to determine the accuracy of impression materials is not indicated.

ISO recommends the use of a travelling microscope for measuring the distance on the specimens. Unfortunately, no travelling microscope was available to the researchers. Therefore, it was decided to use a novel technique of measuring dimensions from digital images from the specimens and not from the specimens themselves. This had the benefit of minimizing temperature loss from the moment of removal from the incubator until the image was taken: it was assumed that making a photograph of a specimen was faster than mounting the specimen on a travelling microscope and manipulating the apparatus to do 3 readings. By the time the 3rd reading is done using a microscope, the specimen must have cooled off. A second benefit was that the specimens are digitally stored and can be measured and re-measured at any given time. It is not known how the results using this digital technique would compare to results using the conventional technique of a travelling microscope.

Since no undercuts were incorporated into the ISO specified mold, no elastic recovery could have played a role in the fluctuations of the %DC. The influence of elastic recovery on dimensional stability could be investigated further.

5.2 LIMITATIONS

Dimensional stability is only one physical property that contributes to the performance of an impression material. In addition, the results of *in vitro* studies do not necessarily predict clinical performance. However, impression materials fall in the category of dental materials where laboratory tests have some level of correlation with clinical performance since impression materials function for a short period of time and predominantly extra-orally (Kelly, 2006).

This study is limited because only 1 elevated temperature, 66°C, was chosen to investigate the dimensional stability of elastomer impression materials.

Another limitation is the use of one brand for each type of elastomer. Extrapolation of the results obtained with these products to other brands of related materials must be done with care. Minor variations in chemical composition may influence results.

5.3 CONCLUSIONS AND RECOMMENDATIONS

Within the limitations of this *in vitro* study, the following conclusions can be made in terms of dimensional stability:

1. Silicone impression material recovers after an extended period at elevated temperature.
2. Polyether impression material did not recover after an extended period at elevated temperature. It showed an expansion, after an initial contraction.
3. For both impression materials, storing at room temperature for 2 weeks resulted in the highest dimensional accuracy.

4. Even though statistical differences were found, both materials are within ISO-specification for dimensional stability, even when heated for a prolonged period.

It is recommended that silicone and polyether impression material be kept at room temperature if they cannot be poured within 30 minutes. This is especially important for polyether impression material, since it does not recover to “pre-heating” dimensional accuracy.

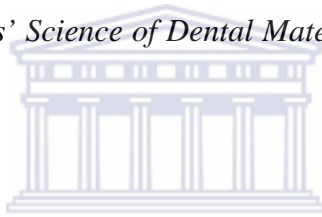


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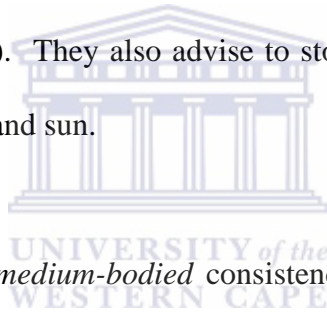
ADDENDA

Addendum 1: Material specifications Affinis & Impregum

AFFINIS®PRECIOUS *regular body* is a silicone-based impression material for use in dentistry with automatic mixing device. It is classified as an addition-type silicone elastomer, ISO 4823, Type 2, medium consistency.

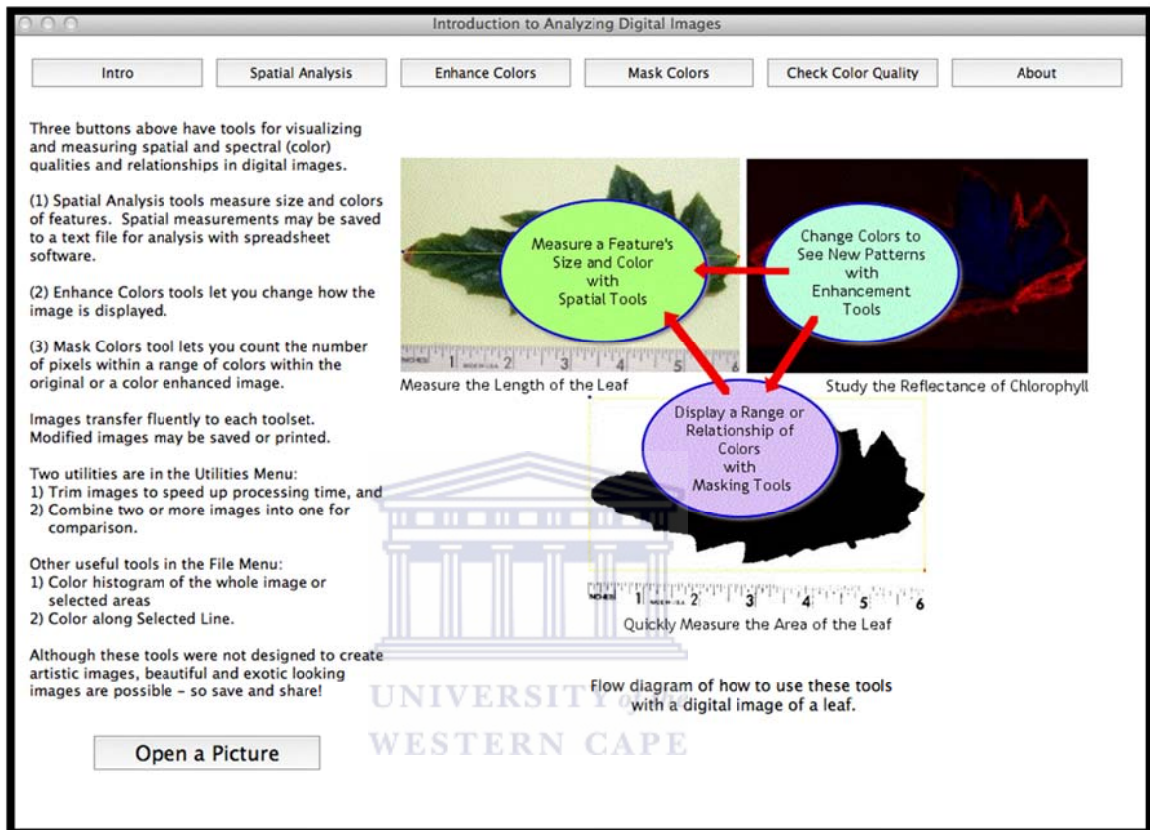
According to the manufacturers, stone models can be poured after 30 minutes at the earliest. The impression remains dimensionally stable for a practically unlimited period of time (for at least one week). They also advise to store impressions at room temperature, and avoid exposure to heat and sun.

Impregum™Penta™ is a *medium-bodied* consistency polyether impression material for the Pentamix mixing device, both products manufactured by 3M ESPE. According to the manufacturers a cast from the impression should be prepared with a specialized stone plaster no earlier than 30 min and no later than 14 days after impression taking.



Addendum 2: Digital measuring sequence using analysingDigitalImaging software version

Fig 1: Opening page of AnalyzingDigitalImages, version 11, August 2008.



A picture needs to be opened and the tab “Spatial Analysis” is selected to start the measuring process.

Fig 2 to 8 indicates how to manually calibrate the pixel size using a ruler with known lengths

Fig 2

Manually Calibrate The Pixel Size

- 1) Click on the beginning of an object of known length visible in the image.
- 2) Drag to the end of the scale. Release the mouse.

TIP: Draw along as much of the scale as possible. The longer the line, the more precise the measurement.

A colored line is drawn on the image. If the line does not match the scale, either redraw the line or fine tune the start and stop positions of the line with the small arrows next to the x and y positions of the line end points, which are located below the image.

- 3) When satisfied with the fit of the line to the scale, enter the length of the scale used in the labeled white box below.
- 4) Enter two letters that represent the unit of the scale being used in the labeled white box below. For example, type "in" for inches and "cm" for centimeters.
- 5) Click 'Done' when finished. To re-run the calibration method click 'Calibrate Length' in the File menu.

Start of Line 81 2243

End of Line 5086 2251

Zoom In Magnification: 0,12 x Zoom Out

Length of Drawn Line

Unit of Length

When zoomed in, pan around the image by using the arrow keys or hold the SHIFT key and click and drag the image.

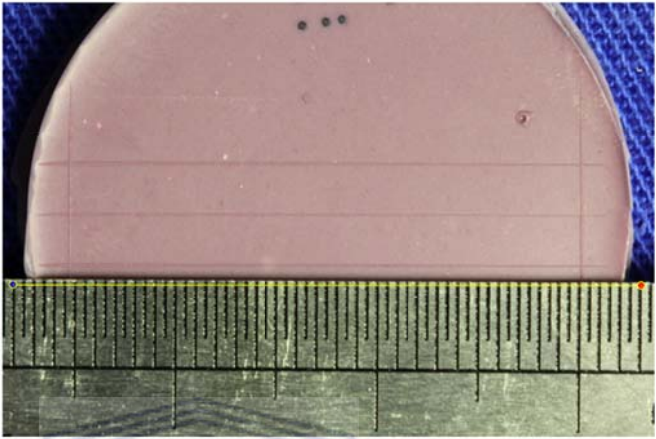


Fig 3

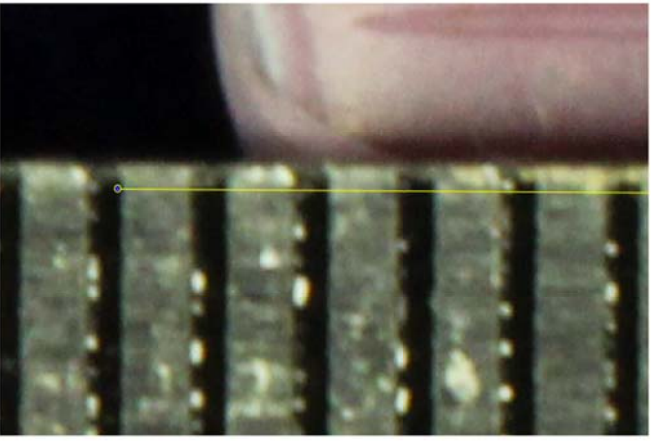
Manually Calibrate The Pixel Size

- 1) Click on the beginning of an object of known length visible in the image.
- 2) Drag to the end of the scale. Release the mouse.



TIP: Draw along as much of the scale as possible. The longer the line, the more precise the measurement.

A colored line is drawn on the image. If the line does not match the scale, either redraw the line or fine tune the start and stop positions of the line with the small arrows next to the x and y positions of the line end points, which are located below the image.

- 3) When satisfied with the fit of the line to the scale, enter the length of the scale used in the labeled white box below.
- 4) Enter two letters that represent the unit of the scale being used in the labeled white box below. For example, type "in" for inches and "cm" for centimeters.
- 5) Click 'Done' when finished. To re-run the calibration method click 'Calibrate Length' in the File menu.



IMG_3772.JPG is 5184 by 3456 pixels

	X	Y	
Start of Line	92	2215	
End of Line	5086	2251	

Length of Drawn Line

Unit of Length

Zoom In Magnification: 1,27 x Zoom Out

When zoomed in, pan around the image by using the arrow keys or hold the SHIFT key and click and drag the image.

Fig 4

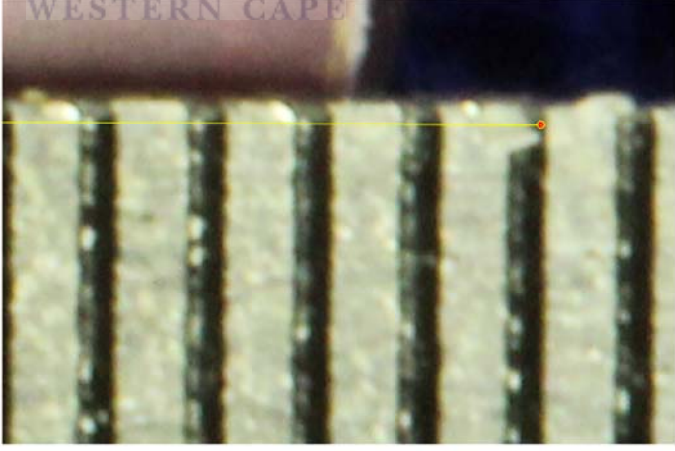
Manually Calibrate The Pixel Size

- 1) Click on the beginning of an object of known length visible in the image.
- 2) Drag to the end of the scale. Release the mouse.

TIP: Draw along as much of the scale as possible. The longer the line, the more precise the measurement.

A colored line is drawn on the image. If the line does not match the scale, either redraw the line or fine tune the start and stop positions of the line with the small arrows next to the x and y positions of the line end points, which are located below the image.



- 3) When satisfied with the fit of the line to the scale, enter the length of the scale used in the labeled white box below.
- 4) Enter two letters that represent the unit of the scale being used in the labeled white box below. For example, type "in" for inches and "cm" for centimeters.
- 5) Click 'Done' when finished. To re-run the calibration method click 'Calibrate Length' in the File menu.



Manually Calibrate the Size of Pixels

WESTERN CAPE

IMG_3772.JPG is 5184 by 3456 pixels

	X	Y	
Start of Line	92	2215	
End of Line	5084	2241	

Length of Drawn Line

Unit of Length

Zoom In Magnification: 1,27 x Zoom Out

When zoomed in, pan around the image by using the arrow keys or hold the SHIFT key and click and drag the image.

Fig 5

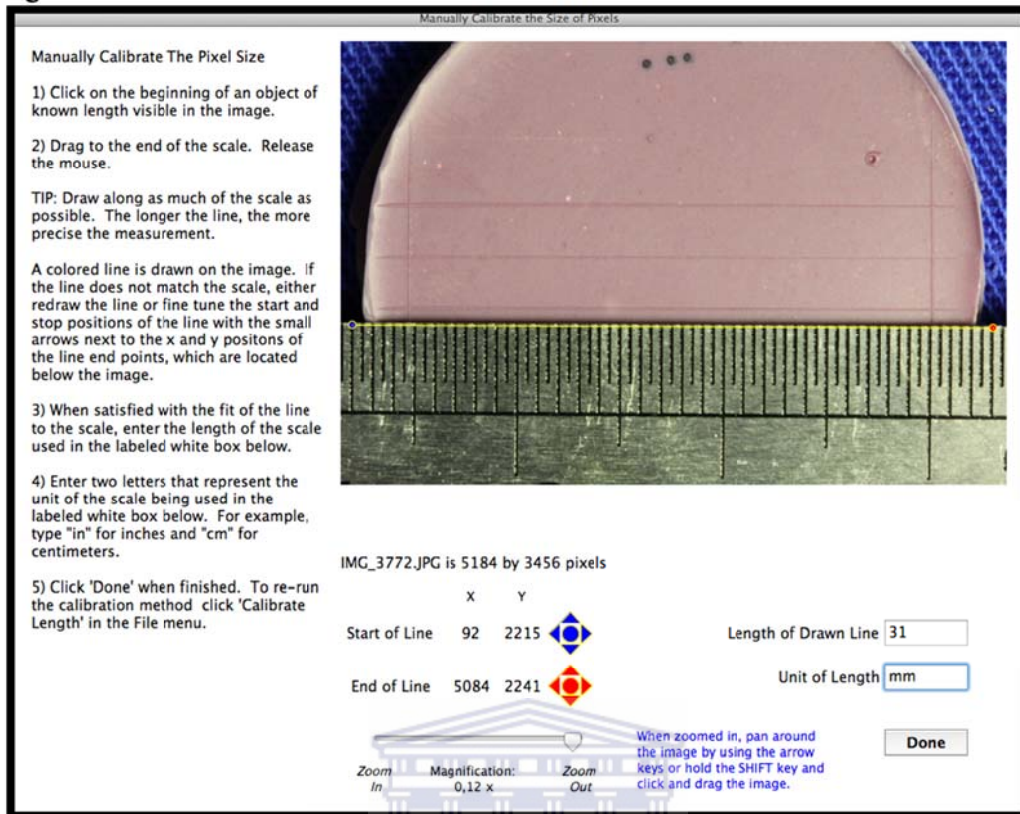


Fig 6

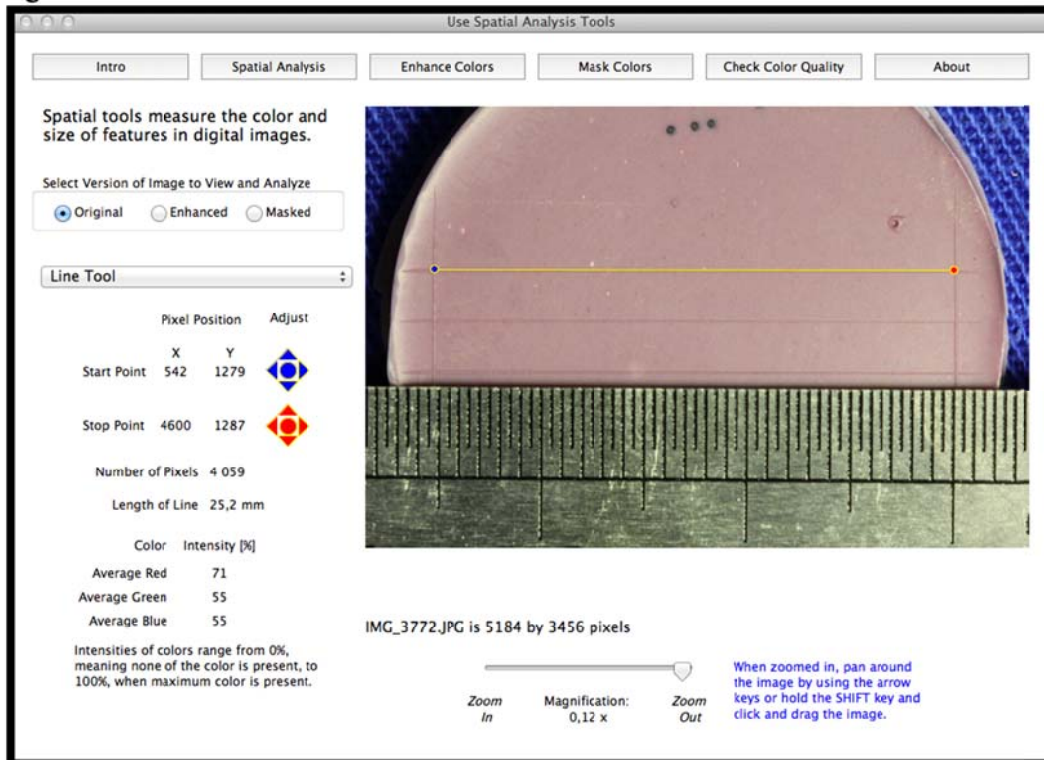
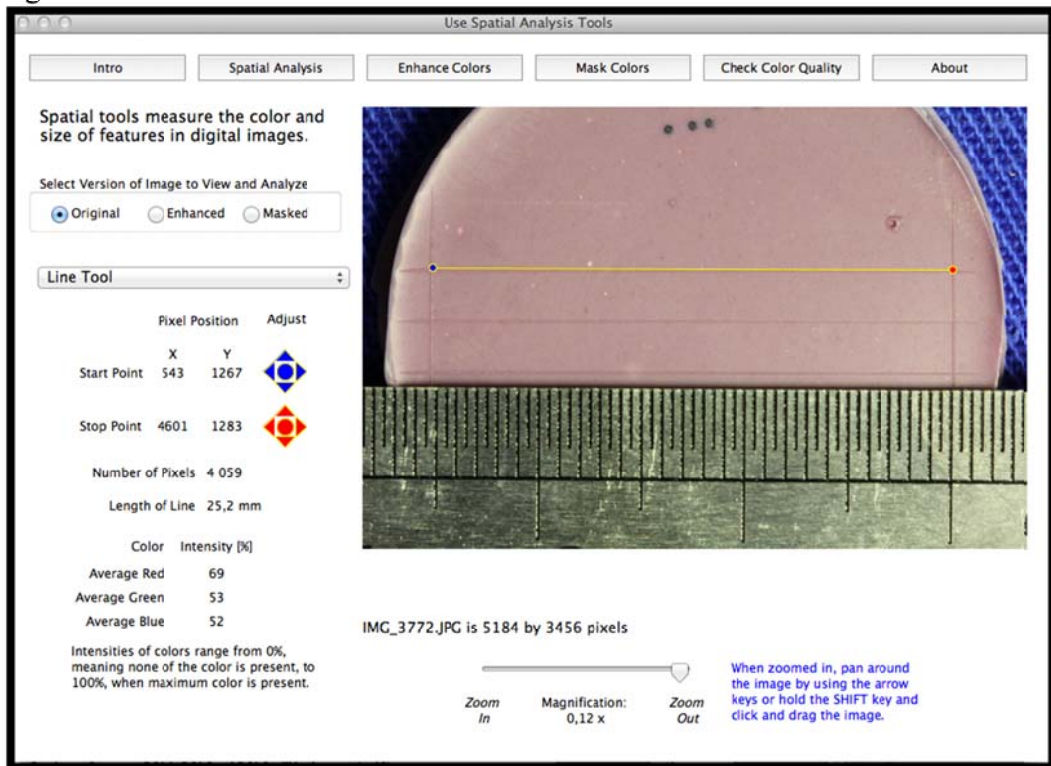


Fig 7



Fig 8



Addendum 3: Data capturing sheet

GROUP S*21*1				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (yes / no)
(i)				
(ii)				
(iii)				
(iv)				
(v)				
*Until n=10				



Addendum 4: Raw data (Dimensions) & %DC

Table used for recording distance

Group S*21*1				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
DSC_0002.JPG 1	25,19546	25,19748	0,038864444	Yes
	25,20069			
	25,19628			
DSC_0003.JPG 2	25,20437	25,19773	0,03784622	Yes
	25,20227			
	25,18656			
DSC_0004.JPG 3	25,21429	25,22870	-0,085001921	Yes
	25,24527			
	25,22654			
DSC_0005.JPG 4	25,24484	25,24484	-0,14903106	Yes
	25,24484			
	25,24484			
DSC_0006.JPG 5	25,24484	25,19130	0,063367954	Yes
	25,17345			
	25,15561			
DSC_0007.JPG 6	25,22464	25,22666	-0,076922243	Yes
	25,23214			
	25,22321			
DSC_0008.JPG 7	25,25365	25,25295	-0,181204314	Yes
	25,25365			
	25,25155			
DSC_0009.JPG 8	25,26349	25,26168	-0,215823951	Yes
	25,26349			
	25,25805			
DSC_0010.JPG 9	25,24370	25,24668	-0,156330541	Yes
	25,24370			
	25,25264			
DSC_0011.JPG 10	25,25474	25,24423	-0,1465979	Yes
	25,23687			
	25,24107			

Group S*21*2				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
DSC_0064.JPG 1	25,15833	25,15532	0,206104534	Yes
	25,14930			
	25,15833			
DSC_0065.JPG 2	25,12695	25,13154	0,300455609	Yes
	25,11792			
	25,14974			
DSC_0066.JPG 3	25,14716	25,14945	0,229391464	Yes
	25,15618			
	25,14501			
DSC_0067.JPG 4	25,14759	25,14902	0,231084097	Yes
	25,14974			
	25,14974			
DSC_0068.JPG 5	25,13953	25,13953	0,268745185	Yes
	25,13953			
	25,13953			
DSC_0069.JPG 6	25,14856	25,14498	0,247124441	Yes
	25,13782			
	25,14856			
DSC_0070.JPG 7	25,16099	25,16156	0,181362998	Yes
	25,16099			
	25,16269			
DSC_0071.JPG 8	25,13953	25,13953	0,268745185	Yes
	25,13953			
	25,13953			
DSC_0072.JPG 9	25,16306	25,15776	0,196424789	Yes
	25,15404			
	25,15618			
DSC_0073.JPG 10	25,14759	25,14601	0,243025095	Yes
	25,14974			
	25,14071			

Group S*21*3				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
IMG_3730.JPG 1	25,20648	25,20441	0,011345932	Yes
	25,20028			
	25,20648			
IMG_3731.JPG 2	25,22323	25,22157	-0,056716434	Yes
	25,21825			
	25,22323			
IMG_3732.JPG 3	25,19573	25,20071	0,026024235	Yes
	25,20475			
	25,20166			
IMG_3733.JPG 4	25,17453	25,18445	0,090555874	Yes
	25,19833			
	25,18048			
IMG_3734.JPG 5	25,20285	25,20317	0,016291594	Yes
	25,20285			
	25,20380			
IMG_3735.JPG 6	25,19952	25,19786	0,037356943	Yes
	25,20048			
	25,19357			
IMG_3736.JPG 7	25,23470	25,23105	-0,094337851	Yes
	25,22970			
	25,22876			
IMG_3737.JPG 8	25,20143	25,20175	0,021924889	Yes
	25,20238			
	25,20143			
IMG_3739.JPG 9	25,20072	25,20072	0,025997787	Yes
	25,20072			
	25,20072			
IMG_3740.JPG 10	25,19357	25,21247	-0,020628967	Yes
	25,25124			
	25,19261			

Group S*66*1				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
DSC_0025.JPG 1	25,25206	25,24534	-0,151027838	Yes
	25,24534			
	25,23863			
DSC_0026.JPG 2	25,24134	25,24492	-0,149335205	Yes
	25,25026			
	25,24315			
DSC_0027.JPG 3	25,23372	25,23145	-0,095898247	Yes
	25,22690			
	25,23372			
DSC_0028.JPG 4	25,23423	25,23273	-0,100976147	Yes
	25,22972			
	25,23423			
DSC_0029.JPG 5	25,22581	25,22622	-0,075176715	Yes
	25,22754			
	25,22532			
DSC_0030.JPG 6	25,24107	25,23949	-0,127820251	Yes
	25,24317			
	25,23424			
DSC_0031.JPG 7	25,23267	25,23039	-0,091706336	Yes
	25,22583			
	25,23267			
DSC_0032.JPG 8	25,19220	25,19377	0,053569195	Yes
	25,19691			
	25,19220			
DSC_0033.JPG 9	25,22846	25,22951	-0,088228503	Yes
	25,23004			
	25,23004			
DSC_0034.JPG 10	25,23793	25,23793	-0,121618337	Yes
	25,23793			
	25,23793			

Group S*66*2				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
DSC_0105.JPG 1	25,14617	25,14327	0,253908197	Yes
	25,14617			
	25,13747			
DSC_0106.JPG 2	25,11491	25,12712	0,317990231	Yes
	25,12980			
	25,13664			
DSC_0107.JPG 3	25,16649	25,15488	0,207850062	Yes
	25,14037			
	25,15778			
DSC_0108.JPG 4	25,14867	25,14936	0,229748504	Yes
	25,15074			
	25,14867			
DSC_0109.JPG 5	25,14244	25,13906	0,270596502	Yes
	25,13372			
	25,14103			
DSC_0110.JPG 6	25,15406	25,15924	0,190540244	Yes
	25,16090			
	25,16277			
DSC_0111.JPG 7	25,10060	25,10185	0,418225851	Yes
	25,10060			
	25,10435			
DSC_0112.JPG 8	25,14721	25,13788	0,275277691	Yes
	25,12980			
	25,13664			
DSC_0113.JPG 9	25,10555	25,09974	0,426583227	Yes
	25,09684			
	25,09684			
DSC_0114.JPG 10	25,11238	25,11176	0,378925025	Yes
	25,11051			
	25,11238			

Group S*66*3				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
IMG_3753.JPG 1	25,23097	25,23006	-0,090383966	Yes
	25,23718			
	25,22202			
IMG_3754.JPG 2	25,22750	25,23371	-0,104890361	Yes
	25,23371			
	25,23993			
IMG_3755.JPG 3	25,23602	25,23395	-0,105829243	Yes
	25,23602			
	25,22981			
IMG_3756.JPG 4	25,22129	25,22206	-0,058660318	Yes
	25,22129			
	25,22360			
IMG_3757.JPG 5	25,25396	25,24774	-0,1605489	Yes
	25,25396			
	25,23531			
IMG_3758.JPG 6	25,24357	25,24941	-0,167173972	Yes
	25,25487			
	25,24980			
IMG_3759.JPG 7	25,22720	25,22590	-0,073880793	Yes
	25,22720			
	25,22329			
IMG_3760.JPG 8	25,21823	25,21654	-0,0367751	Yes
	25,21201			
	25,21939			
IMG_3761.JPG 9	25,21939	25,22016	-0,051136035	Yes
	25,21939			
	25,22171			
IMG_3762.JPG 10	25,22634	25,22726	-0,079289284	Yes
	25,22013			
	25,23531			

Group P*21*1				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
DSC_0015.JPG 1	25,19214	25,20352	0,014909718	Yes
	25,21338			
	25,19979			
	25,20875			
DSC_0016.JPG 2	25,15338	25,14742	0,237457919	Yes
	25,13549			
	25,15338			
DSC_0017.JPG 3	25,16394	25,17600	0,124064721	Yes
	25,18883			
	25,17523			
DSC_0018.JPG 4	25,15731	25,16018	0,186837609	Yes
	25,16161			
	25,16161			
DSC_0019.JPG 5	25,17756	25,17911	0,111740236	Yes
	25,17988			
	25,17988			
DSC_0020.JPG 6	25,16627	25,17446	0,130187293	Yes
	25,18187			
	25,17523			
DSC_0021.JPG 7	25,17954	25,18187	0,100791015	Yes
	25,19083			
	25,17523			
DSC_0022.JPG 8	25,17523	25,18431	0,091098046	Yes
	25,18618			
	25,19152			
DSC_0023.JPG 9	25,18187	25,18563	0,085861462	Yes
	25,19083			
	25,18419			
DSC_0024.JPG 10	25,17756	25,17756	0,117876031	Yes
	25,17756			
	25,17756			

Group P*21*2				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
DSC_0074.JPG 1	25,13383	25,13140	0,301011004	Yes
	25,13813			
	25,12223			
DSC_0075.JPG 2	25,12264	25,11877	0,351089143	Yes
	25,10889			
	25,12479			
DSC_0076.JPG 3	25,11105	25,10732	0,396525765	Yes
	25,10889			
	25,10202			
DSC_0077.JPG 4	25,10964	25,10663	0,39926307	Yes
	25,10061			
	25,10964			
DSC_0078.JPG 5	25,12673	25,12738	0,316958783	Yes
	25,12673			
	25,12867			
DSC_0079.JPG 6	25,09825	25,10126	0,420566445	Yes
	25,09825			
	25,10728			
DSC_0080.JPG 7	25,09770	25,10300	0,413650452	Yes
	25,09770			
	25,11361			
DSC_0081.JPG 8	25,10202	25,09829	0,432348759	Yes
	25,09083			
	25,10202			
DSC_0082.JPG 9	25,11105	25,11105	0,381728448	Yes
	25,11105			
	25,11105			
DSC_0083.JPG 10	25,14772	25,14929	0,230039425	Yes
	25,15242			
	25,14772			

Group P*21*3				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
IMG_3742.JPG 1	25,25035	25,25264	-0,17997451	Yes
	25,25628			
	25,25129			
IMG_3743.JPG 2	25,21782	25,22083	-0,053793998	Yes
	25,22092			
	25,22376			
IMG_3744.JPG 3	25,20854	25,20625	0,004072899	Yes
	25,19668			
	25,21352			
IMG_3745.JPG 4	25,19573	25,20174	0,021938113	Yes
	25,20475			
	25,20475			
IMG_3746.JPG 5	25,21860	25,21923	-0,047446623	Yes
	25,21955			
	25,21955			
IMG_3747.JPG 6	25,19715	25,19850	0,034817993	Yes
	25,19715			
	25,20119			
IMG_3748.JPG 7	25,19905	25,19842	0,035135362	Yes
	25,19810			
	25,19810			
IMG_3749.JPG 8	25,19799	25,19631	0,043505962	Yes
	25,19799			
	25,19294			
IMG_3750.JPG 9	25,21309	25,21515	-0,031260819	Yes
	25,20804			
	25,22433			
IMG_3751.JPG 10	25,26348	25,26206	-0,2173579	Yes
	25,26479			
	25,25792			

Group P*66*1				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
DSC_0035.JPG 1	25,24961	25,24100	-0,133797362	Yes
	25,24120			
	25,23219			
DSC_0036.JPG 2	25,19185	25,18585	0,084988698	Yes
	25,18285			
	25,18285			
DSC_0037.JPG 3	25,25732	25,25732	-0,19854058	Yes
	25,25732			
	25,25732			
DSC_0038.JPG 4	25,16340	25,16040	0,185938397	Yes
	25,14756			
	25,17025			
DSC_0039.JPG 5	25,14921	25,14621	0,242244897	Yes
	25,14921			
	25,14021			
DSC_0040.JPG 6	25,05263	25,05421	0,607218922	Yes
	25,05500			
	25,05500			
DSC_0041.JPG 7	25,15586	25,15286	0,215863622	Yes
	25,15586			
	25,14686			
DSC_0042.JPG 8	25,10188	25,10109	0,42122763	Yes
	25,09288			
	25,10852			
DSC_0043.JPG 9	25,11558	25,12679	0,319299377	Yes
	25,13356			
	25,13122			
DSC_0044.JPG 10	25,13514	25,13800	0,274814862	Yes
	25,13943			
	25,13943			

Group P*66*2				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
DSC_0095.JPG 1	25,10264	25,12137	0,340801107	Yes
	25,13166			
	25,12980			
DSC_0096.JPG 2	25,13539	25,13760	0,276388482	Yes
	25,14203			
	25,13539			
DSC_0097.JPG 3	25,13850	25,13270	0,295840539	Yes
	25,12980			
	25,12980			
DSC_0098.JPG 4	25,12857	25,12402	0,330288269	Yes
	25,12174			
	25,12174			
DSC_0099.JPG 5	25,10643	25,10214	0,417062165	Yes
	25,10435			
	25,09565			
DSC_0100.JPG 6	25,12231	25,11940	0,348589865	Yes
	25,12231			
	25,11359			
DSC_0101.JPG 7	25,15033	25,15385	0,211922961	Yes
	25,15033			
	25,16090			
DSC_0102.JPG 8	25,11343	25,10763	0,395309185	Yes
	25,09602			
	25,11343			
DSC_0103.JPG 9	25,14161	25,14673	0,240195224	Yes
	25,15032			
	25,14825			
DSC_0104.JPG 10	25,15738	25,15517	0,206712824	Yes
	25,15738			
	25,15074			

Group P*66*3				
Specimen	Measurement (mm)	Average (L1)	DC (%)	Pass (Yes/No)
IMG_3764.JPG 1	25,22433	25,23054	-0,092288178	Yes
	25,23674			
	25,23054			
IMG_3765.JPG 2	25,23559	25,23766	-0,120533994	Yes
	25,23559			
	25,24179			
IMG_3766.JPG 3	25,30536	25,30951	-0,405583997	Yes
	25,31781			
	25,30536			
IMG_3767.JPG 4	25,25335	25,25880	-0,204411901	Yes
	25,26087			
	25,26218			
IMG_3768.JPG 5	25,28732	25,29352	-0,342163148	Yes
	25,29973			
	25,29352			
IMG_3769.JPG 6	25,25474	25,25889	-0,204755717	Yes
	25,26096			
	25,26096			
IMG_3770.JPG 7	25,31964	25,32007	-0,447476667	Yes
	25,32093			
	25,31964			
IMG_3772.JPG 8	25,27199	25,27777	-0,279654735	Yes
	25,28311			
	25,27820			
IMG_3773.JPG 9	25,30666	25,30502	-0,387784901	Yes
	25,30666			
	25,30175			
IMG_3774.JPG 10	25,26316	25,26774	-0,239864632	Yes
	25,27428			
	25,26577			

Addendum 5: Multiple comparisons

LSD test; variable %DC (Spreadsheet10)

Simultaneous confidence intervals

Effect: material*temperature*time

1st - Mean	2nd - Mean	Mean - Differ.	Standard - Error	p	-95.00% - Cnf.Lmt	+95.00% - Cnf.Lmt
P*21*1	P*21*2	-0.244235	0.042041	0.000000	-0.328043	-0.160428
P*21*1	P*21*3	0.159119	0.042041	0.000316	0.075311	0.242926
P*21*1	P*66*1	-0.081843	0.044842	0.072125	-0.171234	0.007547
P*21*1	P*66*2	-0.186228	0.044842	0.000089	-0.275619	-0.096838
P*21*1	P*66*3	0.392534	0.044842	0.000000	0.303143	0.481924
P*21*1	S*21*1	0.207166	0.044842	0.000016	0.117775	0.296556
P*21*1	S*21*2	-0.117164	0.044842	0.010925	-0.206554	-0.027773
P*21*1	S*21*3	0.114301	0.044842	0.012935	0.024910	0.203691
P*21*1	S*66*1	0.214904	0.044842	0.000009	0.125514	0.304295
P*21*1	S*66*2	-0.176882	0.044842	0.000184	-0.266272	-0.087491
P*21*1	S*66*3	0.212939	0.044842	0.000010	0.123548	0.302329
P*21*2	P*21*3	0.403354	0.042041	0.000000	0.319546	0.487162
P*21*2	P*66*1	0.162392	0.044842	0.000542	0.073002	0.251783
P*21*2	P*66*2	0.058007	0.044842	0.199941	-0.031383	0.147398
P*21*2	P*66*3	0.636769	0.044842	0.000000	0.547379	0.726160
P*21*2	S*21*1	0.451401	0.044842	0.000000	0.362010	0.540791
P*21*2	S*21*2	0.127072	0.044842	0.005965	0.037681	0.216462
P*21*2	S*21*3	0.358536	0.044842	0.000000	0.269146	0.447927
P*21*2	S*66*1	0.459139	0.044842	0.000000	0.369749	0.548530
P*21*2	S*66*2	0.067354	0.044842	0.137464	-0.022037	0.156744
P*21*2	S*66*3	0.457174	0.044842	0.000000	0.367784	0.546565
P*21*3	P*66*1	-0.240962	0.044842	0.000001	-0.330352	-0.151571
P*21*3	P*66*2	-0.345347	0.044842	0.000000	-0.434738	-0.255957
P*21*3	P*66*3	0.233415	0.044842	0.000002	0.144025	0.322806
P*21*3	S*21*1	0.048047	0.044842	0.287534	-0.041344	0.137437
P*21*3	S*21*2	-0.276282	0.044842	0.000000	-0.365673	-0.186892
P*21*3	S*21*3	-0.044818	0.044842	0.320918	-0.134208	0.044573
P*21*3	S*66*1	0.055785	0.044842	0.217516	-0.033605	0.145176
P*21*3	S*66*2	-0.336001	0.044842	0.000000	-0.425391	-0.246610
P*21*3	S*66*3	0.053820	0.044842	0.233985	-0.035570	0.143211
P*66*1	P*66*2	-0.104385	0.042041	0.015362	-0.188193	-0.020577
P*66*1	P*66*3	0.474377	0.042041	0.000000	0.390569	0.558185
P*66*1	S*21*1	0.289009	0.044842	0.000000	0.199618	0.378399
P*66*1	S*21*2	-0.035320	0.044842	0.433476	-0.124711	0.054070
P*66*1	S*21*3	0.196144	0.044842	0.000040	0.106754	0.285535
P*66*1	S*66*1	0.296747	0.044842	0.000000	0.207357	0.386138
P*66*1	S*66*2	-0.095039	0.044842	0.037504	-0.184429	-0.005648
P*66*1	S*66*3	0.294782	0.044842	0.000000	0.205392	0.384173
P*66*2	P*66*3	0.578762	0.042041	0.000000	0.494955	0.662570
P*66*2	S*21*1	0.393394	0.044842	0.000000	0.304003	0.482784
P*66*2	S*21*2	0.069065	0.044842	0.127899	-0.020326	0.158455
P*66*2	S*21*3	0.300529	0.044842	0.000000	0.211139	0.389920
P*66*2	S*66*1	0.401132	0.044842	0.000000	0.311742	0.490523

P*66*2	S*66*2	0.009346	0.044842	0.835480	-0.080044	0.098737
P*66*2	S*66*3	0.399167	0.044842	0.000000	0.309777	0.488558
P*66*3	S*21*1	-0.185368	0.044842	0.000095	-0.274759	-0.095978
P*66*3	S*21*2	-0.509698	0.044842	0.000000	-0.599088	-0.420307
P*66*3	S*21*3	-0.278233	0.044842	0.000000	-0.367623	-0.188842
P*66*3	S*66*1	-0.177630	0.044842	0.000173	-0.267020	-0.088239
P*66*3	S*66*2	-0.569416	0.044842	0.000000	-0.658806	-0.480025
P*66*3	S*66*3	-0.179595	0.044842	0.000149	-0.268985	-0.090204
S*21*1	S*21*2	-0.324329	0.042041	0.000000	-0.408137	-0.240522
S*21*1	S*21*3	-0.092865	0.042041	0.030365	-0.176672	-0.009057
S*21*1	S*66*1	0.007738	0.044842	0.863471	-0.081652	0.097129
S*21*1	S*66*2	-0.384047	0.044842	0.000000	-0.473438	-0.294657
S*21*1	S*66*3	0.005773	0.044842	0.897913	-0.083617	0.095164
S*21*2	S*21*3	0.231465	0.042041	0.000001	0.147657	0.315272
S*21*2	S*66*1	0.332068	0.044842	0.000000	0.242677	0.421458
S*21*2	S*66*2	-0.059718	0.044842	0.187143	-0.149109	0.029672
S*21*2	S*66*3	0.330103	0.044842	0.000000	0.240712	0.419493
S*21*3	S*66*1	0.100603	0.044842	0.027941	0.011213	0.189994
S*21*3	S*66*2	-0.291183	0.044842	0.000000	-0.380573	-0.201792
S*21*3	S*66*3	0.098638	0.044842	0.031040	0.009248	0.188029
S*66*1	S*66*2	-0.391786	0.042041	0.000000	-0.475594	-0.307978
S*66*1	S*66*3	-0.001965	0.042041	0.962849	-0.085773	0.081843
S*66*2	S*66*3	0.389821	0.042041	0.000000	0.306013	0.473629



Addendum 6: Descriptive Statistics

Effect	Descriptive Statistics (Spreadsheet10)									
	Level of Factor	Level of Factor	Level of Factor	N	%DC Mean	%DC Std.Dev.	%DC Std.Err	%DC -95.00%	%DC +95.00%	
Total				120	0.078971	0.217153	0.019823	0.039719	0.118223	
material	P			60	0.113631	0.248617	0.032096	0.049406	0.177855	
material	S			60	0.044311	0.175661	0.022678	-0.001067	0.089689	
temperature	21			60	0.100324	0.174444	0.022521	0.055260	0.145387	
temperature	66			60	0.057618	0.252492	0.032597	-0.007608	0.122843	
time	1			40	0.035132	0.185570	0.029341	-0.024217	0.094480	
time	2			40	0.301315	0.079066	0.012501	0.276029	0.326602	
time	3			40	-0.099535	0.133566	0.021119	-0.142252	-0.056818	
material*temperature	P	21		30	0.148560	0.182890	0.033391	0.080268	0.216853	
material*temperature	P	66		30	0.078701	0.299631	0.054705	-0.033183	0.190585	
material*temperature	S	21		30	0.052087	0.153778	0.028076	-0.005335	0.109509	
material*temperature	S	66		30	0.036534	0.197496	0.036058	-0.037212	0.110280	
material*time	P	1		20	0.161110	0.175565	0.039258	0.078943	0.243277	
material*time	P	2		20	0.335420	0.073274	0.016384	0.301127	0.369713	
material*time	P	3		20	-0.155638	0.158969	0.035547	-0.230038	-0.081238	
material*time	S	1		20	-0.090847	0.080314	0.017959	-0.128435	-0.053259	
material*time	S	2		20	0.267211	0.070811	0.015834	0.234070	0.300352	
material*time	S	3		20	-0.043432	0.068711	0.015364	-0.075590	-0.011274	
temperature*time	21	1		20	0.016605	0.133432	0.029836	-0.045843	0.079053	
temperature*time	21	2		20	0.300888	0.082701	0.018492	0.262183	0.339593	
temperature*time	21	3		20	-0.016522	0.075892	0.016970	-0.052040	0.018997	
temperature*time	66	1		20	0.053658	0.228382	0.051068	-0.053228	0.160544	
temperature*time	66	2		20	0.301743	0.077409	0.017309	0.265515	0.337972	
temperature*time	66	3		20	-0.182548	0.127873	0.028593	-0.242395	-0.122702	
material*temperature*time	P	21	1	10	0.120188	0.059507	0.018818	0.077619	0.162757	
material*temperature*time	P	21	2	10	0.364424	0.064329	0.020343	0.318406	0.410442	
material*temperature*time	P	21	3	10	-0.038931	0.091504	0.028936	-0.104388	0.026527	
material*temperature*time	P	66	1	10	0.202031	0.240434	0.076032	0.030035	0.374028	
material*temperature*time	P	66	2	10	0.306417	0.072987	0.023080	0.254205	0.358628	
material*temperature*time	P	66	3	10	-0.272346	0.121282	0.038353	-0.359105	-0.185586	
material*temperature*time	S	21	1	10	-0.086977	0.101006	0.031941	-0.159233	-0.014722	
material*temperature*time	S	21	2	10	0.237352	0.036468	0.011532	0.211264	0.263440	
material*temperature*time	S	21	3	10	0.005887	0.051674	0.016341	-0.031078	0.042852	
material*temperature*time	S	66	1	10	-0.094716	0.058153	0.018390	-0.136316	-0.053116	
material*temperature*time	S	66	2	10	0.297070	0.085290	0.026971	0.236057	0.358083	
material*temperature*time	S	66	3	10	-0.092751	0.043492	0.013753	-0.123863	-0.061639	