Colour stability of three modern ceramic materials after repeated firing.

BY

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I, Ansuya Naidoo, do declare that this dissertation is representative of my own work in both conception and execution (except where acknowledgements indicate to the contrary).

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DEDICATION

***This study is dedicated to the supreme power, through which all things are made possible, my creator. To Thee, I offer my prostrations and salutations*.**

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ABSTRACT

Background

Shade matching is a challenging aspect of aesthetic dentistry (Vichi *et al*., 2011). For a long time, aesthetically pleasing prostheses have been achieved using the porcelain-fused-to-metal crown whereby the metal is masked with an opaque layer and then consecutive layers of veneering ceramic are built up (McLean, 1979). The growing popularity of all-ceramic restorations has created a need for an understanding of its colour characteristics in preference to porcelain fused to metal restorations. The study aims to assist dental technicians in making a more informed decision when choosing an all-ceramic system in terms of colour stability.

Objectives

The objectives were to identify colour changes of the zirconia after each firing cycle and defining if there are any microstructural changes in the zirconia cores after subsequent firing.

Method

This is a quantitative study with a sample size of eighteen which was used for each experimental group. Green state zirconia blocks from Cercon (Dentsply DeguDent, USA), Lava (3M ESPE,USA), and Zirkon Zahn (Zirkonzahn Gmbh, Bruneck, Italy) were trimmed, finished and sintered to a final thickness of 0.5mm according to the manufacturers' instructions. Each sample group was obtained directly from the manufacturer. In each group, nine specimens were shaded and nine were unshaded. The colour of each specimen was recorded before being exposed to firing cycles. This data formed the control group. All specimens were subjected to three firings and spectrophotometer reading. After each firing, SEM analysis was done after one and three firings. There was a total of thirty six specimens x three firings; n = 108.

The raw data for the statistical analysis was obtained from the spectrophotometer readings recorded after the firings (n=108). The data of the study was analyzed using the SPSS version 22.0. The data were compared among the three groups using Pearsons chi square tests, where the data of the firings were measured as a binary or nominal variable, and using t-tests where a measured normally distributed variable was compared. The general linear model (GLM) looked at combinations of the variables and their effects on the dependant variable. The Multivariate tests table indicates the actual result of the one-way MANOVA.

Results

The Cercon results for both the control and experimental groups reveal the same result patterns and was maintained before and after firing. Lava also revealed the same results before and after firing, however, the cores in the control group differed in colour. The Zirkon Zahn system proved to be the least stable in colour.

Conclusion

This study supports the hypothesis that colour differences would occur relative to the number of firings for shaded zirconia. It can be concluded that Cercon displayed the most stable results in terms of colour. The acumen gained in this study may better assist dental technicians in their selection of an all-ceramic zirconia system.

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DEFINITIONS

a*	In CIELAB opponent-color scale, the degree of redness/greenness (Sahin et *al*., 2010)
Bridge	A non-removable dental prosthesis which replaces missing teeth (Sahin et *al*., 2010)
b*	In CIELAB opponent-colour scale, the degree of blueness/yellowness (Sahin et *al.*, 2010)
C(Chroma, Saturation)	The attribute of colour perception determines the degree of saturation of particular hue (Anusavice, 2007)
CAD-CAM	A ceramic that is formulated for the production of the whole or part of an all-ceramic prosthesis through the use of a computer-aided design and computer-aided manufacturing process (Anusavice, 2003)
Colour	Sensation induced from light of varying wavelengths reaching the eye (Anusavice, 2003)
Copy-milling	The process of cutting or grinding a structure using a device that traces the surface of a master metal, ceramic, or polymer pattern and transfers the traced spatial positions to a cutting station where a blank is cut or ground in a manner similar to a key-cutting procedure (Owen et *al*., 2012)
Connector	A connector is the part of a bridge which connects the pontic (false tooth) to the retainer (crown portion of the bridge) (Owen et *al.*, 2012)
Core ceramic	An opaque dental ceramic material that provides sufficient strength toughness and stiffness to support the overlying layers of veneering ceramics (Anusavice, 2003)
Feldspar	An important constituent of dental porcelain (Vagkopoulou et *al.*, 2009)
Final restoration	Finished product e.g. A crown ready to be inserted in the mouth (Ozturk et *al.*, 2008)

Flexural strength	(Bending strength or Modulus of rupture)-Force per unit area at the point of fracture of a test specimen subjected to flexural loading (Ozturk et *al.*, 2008)
Fluorescence	The process whereby electromagnetic radiation of one spectral region is absorbed and reradiated at other, usually longer, wavelengths (Vagkopoulou et *al.*, 2009)
Frit	A ceramic composition that has been fused in a fusing oven and cooled to form glass and then ground further into a fine powder (Vagkopoulou et *al.*, 2009)
Gloss	The property of a surface which involves specular reflection and responsible for a mirror-like or lustrous appearance (Vichi et *al.*, 2011)
Grain size	Also known as particle size, refers to the diameter of individual grains of sediment, or the lithified particles in clastic rocks (Kim et *al*., 2013)
Green state	A term referring to an as-pressed condition before sintering (Owen et *al*., 2012)
H (Hue)	Dominant color of an object, for example, red, green or blue (Vichi et *al.*, 2011)
L*(Lightness, Brightness, Value)	Relative lightness or darkness of a colour (Anusavice, 2003).
Metal-ceramic prosthesis	A partial crown, full crown or fixed partial denture made with a metal substrate to which porcelain is bonded for aesthetic enhancement via an intermediate metal oxide layer. The terms porcelain-fused-to-metal (PFM), porcelain-bonded-to-metal (PBM), porcelain-to-metal (PTM), and ceramometal are also used to describe these prostheses, but metal-ceramic is the preferred term (Anusavice, 2003)
Metamerism	Phenomenon in which the color of an object under one type of light appears to change when illuminated by a different light source (Anusavice, 2003)
Mouth simulator	An apparatus that simulates the atmosphere of the oral environment (Anusavice, 2003)

Opacity The property of a material to hide what is behind it (Vagkopoulou et *al.*, 2009)

Opalescence The process whereby a material appears yellowish-red in transmitted light and blue in scattered light perpendicular to the transmitted light. This phenomenon was so-called after the appearance of opal stone (Vagkopoulou et *al.*, 2009)

Pontic A pontic is the prosthetic (false) part of the bridge and replaces the missing tooth or teeth (Anusavice, 2003)

Scattering The process by which light passing through granular, fibrous or rough surface matter is redirected throughout a range of angles (Vichi et *al.*, 2011)

Sintering It is a process of forming a solid mass of material by heat and/or pressure without melting it to a point of liquification (Kim et *al.*, 2013)

Spectrophotometer A device that delivers illuminating light (Vichi et *al.*, 2011)

Stain ceramic A mixture of one or more pigmented metal oxides and a low-fusing glass that can modify the shade of the ceramic-based restoration when it is dispersed in an aqueous slurry or monomer medium, applied to the surface of porcelain or other dental ceramic and heated to its vitrification temperature for a specific time (Anusavice, 2003)

Translucency The property of a material by which a major portion of the transmitted light undergoes scattering (Kim et *al.*, 2013)

Transparency The property of a material by which a negligible portion of the transmitted light undergoes scattering(Vagkopoulou et *al.*, 2009)

Zirconia It is a white powdered metal used to create dental frameworks for crowns, bridges and other dental substructures (Kim et *al.*, 2013)

ABBREVIATIONS

CAD	Computer aided design
CAM	Computer aided milling
CCD	Charge-coupled device
CTE	Co-efficient of thermal expansion
FPD	Fixed partial denture
N	Number of subjects in the sample
P	Probability
SEM	Scanning electron microscope
Sig	Significance
Y-TZP	Yttrium - stabilized tetragonal zirconia polycrystals

CHAPTER ONE

INTRODUCTION

1.1 Introduction

Selecting an accurate shade is a challenging aspect of aesthetic dentistry. Achieving a close shade match of the artificial restoration with the natural dentition is a challenge due to the variety of natural tooth colours. Dental practitioners need an understanding and knowledge of colour, light and associated characteristics which contribute to successful shade matching. This includes the light source in which the shade is chosen, the colour of the surrounding walls and furniture in the practitioners' office, the colour of the patients clothing and make-up as well as individuals perception of colour. According to studies by Behrens (2008), "Colour is a sensory impression resulting from the absorption of light with a specific wavelength by receptors on the retina, the light sensitive layer at the back of the eyeball which receives signals and transfers them as nervous impulses to the brain". The issue of metamerism may also be considered a contributing factor. It is therefore necessary to understand the nature of these materials, how they behave, and how long they last in order to achieve an aesthetically pleasing result. Obtaining a natural looking ceramic crown entails two crucial steps. The first being the selection of a shade using a shade guide and the second being the selection of an appropriate material to reproduce this shade (Vichi *et al.,* 2011). The selected shade needs to harmoniously integrate itself with the surrounding oral tissues on the final ceramic crown.

Shade matching has been successfully achieved using the porcelain-fused-to-metal system whereby metal is masked with an opaque layer and then covered by ceramic layers (McLean, 1979). Water is mixed with the selected shade of porcelain powder corresponding to the shade of dentin base, applied to the metal, and then fired. Firing or sintering is a process during which the crown is exposed to increasing temperatures just below that of the melting point of the core metal (McLean, 1979). The firing enables the wet ceramic mixture to harden around the metal. The firing results in shrinkage of the porcelain which is compensated for by a slight overbuild of the porcelain layers. Further layers are applied to mimic the translucency of natural teeth. Porcelain can be veneered onto either metal, gold, aluminium oxide, zirconium oxide or zirconia cores (McLean, 1979).

The introduction of new materials that provide an improved aesthetic appearance with the absence of a metallic structure, such as zirconia have become the treatment of choice for many patients (Denry *et al.,* 2008). Recently, partially stabilized zirconia has received much attention as a core material because of its high flexural strength (Sahin *et al*., 2010). However, due to an increased crystalline content to ensure increased strength, obtaining a desired level of translucency can be difficult (Sahin *et al*., 2010). A higher core translucency results in improved colour visibility of the deeper layers and improved aesthetics (Pecho *et al*., 2012). Translucency is also considered one of the vital factors responsible for matching colour of dental ceramics to what was obtained (Vichi *et al*., 2011). In an attempt to manipulate light transmission, and thus the value, various stained cores are now available. The colour of the core acts to mimic the colour of the dentine. Although the character and composition, of the veneering layer can aid this process, the core still plays a major role. This core veneering complex is subject to various firing cycles. Temperature cycles for layered ceramics can influence the colour stability of the zirconia core to a less significant extent than that of the veneering layers (Sahin *et al.*, 2010). The effect of repeated firings to temperatures up to 800°C on a zirconia core as implemented for ceramic dental core-veneer restorations has not been entirely established (Celik *et al.,* 2008). The influence that these firing cycles have on the final prosthesis is not widely documented (Sahin *et al.,* 2010). To ensure predictability, the colour of the final product needs to be constant. The thermal mismatch between the core and the veneering porcelain is one of the critical factors in ensuring colour stability (Sahin *et al.*, 2010).

There are several zirconia based systems on the market, with varying handling properties and temperature guidelines namely: Lava, Cercon and Zirkon Zahn.

This research study compared the colour stability between the Lava, Cercon, Zirkon Zahn zirconia based systems, in relation to colour consistency of the core material and veneering porcelain, after repeated sintering cycles.

If the colour of zirconia is affected after numerous firings, the results of this study would assist ceramists to make decisions in their choice of an all-ceramic system. A spectrophotometer was used to establish the shades in the different sample groups and micro-structural changes were detected using the scanning electron microscope (SEM).

1.2 The Problem Statement

Limited literature is available on the effect of repeated firings to temperatures up to 800°C on the colour stability of zirconia cores. Proper shade selection is important in gaining aesthetically pleasing results.

1.3 Aim of the Study

The purpose of this study was to evaluate the effect of repeated firings on zirconia ceramics and to compare colour stability after subsequent firing among Lava, Cercon and the Zirkon Zahn systems.

1.4 Objectives of the Study

To validate the colour of the fired zirconia cores with reference to the zirconia cores in the control group in order to establish the stability of each zirconia system.

To identify the colour changes of the zirconia after each firing cycle and determine whether the colour changes are statistically significant.

To demonstrate any microstructural changes in the zirconia cores after subsequent firing.

1.5 Hypothesis

Hypothesis One

It is hypothesized that heating is an aging accelerator and colour stability after subsequent firing is not maintained.

Hypothesis Two

It is hypothesized that heating causes grain growth.

1.6 Delimitations

a) This study measured the effect of three firing cycles on the shade of the zirconia cores and did not investigate the effects of:

- varying core thickness
- different sintering cycles
- varying sintering temperatures

b) These zirconia systems are:

- Lava
- Cercon
- Zirkon Zahn

They were selected based on availability, cost and popularity.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

This review provides insight into the complexity of matching prosthetic restorations to natural tooth shade, with particular reference to zirconia restorations. In addition, it describes the growing popularity of all-ceramic restorations as well as the possible reasons for the increased use of all ceramic systems in preference to porcelain fused to metal restorations (Durand *et al*., 2012). In order to manufacture an aesthetically pleasing crown, the correct shade has to be selected and then reproduced in the restoration. Douglas *et al.* (2007) found that the ability to reproduce the colour of the selected shade differed among laboratories. However, they were still above the clinical threshold for an acceptable shade match. Factors such as numerous firing cycles and type of zirconia selected may contribute to the acceptability of the final crown. Most manufacturers of zirconia based ceramic systems advise the use of a specific veneering porcelain. The Lava system is one such system which calls for the zirconia copings to be layered using the LavaCeram.

In addition, this review deals specifically with the effects of numerous sintering cycles of between two to five times that the zirconia systems are exposed to (Siavikisa *et al*., 2011; Tang *et al*., 2012). The review will then continue to describe colour stability of the zirconia after each firing cycle. The metallurgy of zirconia is analyzed, specifically in its two purest forms in conjunction with its history.

The final section evaluates instrumentation for assessment of colour used historically, and offers motivation for the use of certain instrumentation as opposed to visual analysis using the human eye. Various types of formulae and colour charts previously used by researchers are reviewed in terms of their advantages and disadvantages in relation to this study.

2.2 Brief history of core materials

The end of the 18th century brought the development of dental ceramics together with the development of porcelain dentures (Owen, 2012). Later, at the turn of the nineteenth century, individual porcelain teeth with platinum pins (for retention to metal frameworks) were developed (Owen, 2012).

It was in the 19th century that porcelain was used as a restorative material on natural teeth, and the earliest porcelain jacket crowns were attempted (Schmidseder *et al.*, 2000). In 1896, Dr. Charles Land developed a method of constructing a metal-free jacket crown by firing the ceramic on shaped platinum foil (Schmidseder *et al.*, 2000). These crowns exhibited excellent aesthetics but the low flexural strength of porcelain resulted in a high incidence of failure (Anusavice, 2003). The porcelain material was later replaced with kaolin-free feldspar frit which is a ceramic structure bonded in a special fusing oven and subsequently quenched to form glass (Schmidseder *et al.*, 2000). Since then, feldspathic porcelains with reliable chemical bonding have been used in metal-ceramic prostheses for over 35 years. However, feldspathic porcelains have been too weak to use consistently in the construction of all-ceramic crowns in the absence of a metal foil or cast-metal coping (Anusavice, 2003). Furthermore, their firing shrinkage causes significant discrepancies in fit and adaptation of margins.

What was learnt from the use of the porcelain jacket crown was that porcelain is strong in compression stress, and weak in tension stress. McLean (1976), revealed that porcelain strength depended on even thickness. Tensile stresses were introduced due to uneven thickness within the porcelain material which allowed microcracks to propagate with ease (Owen, 2012; Beck *et al.*, 2010). The even thickness of the porcelain was established as an important factor in the success of a porcelain restoration. Therefore, tooth reduction for the preparation of a porcelain restoration had to also be even (McLean *et al.*, 1976).

The early 1900's saw the first attempts to improve the strength of a porcelain jacket crown. It was the twin-foil technique, in which two platinum foils instead of one were used during the firing of the porcelain (McLean *et al.*, 1976)

One of the platinum foils was to be left in situ to be tin-plated to improve the bonding to glass ionomer cements (Owen, 2012). In the 1960's, porcelain was fused to metal to enhance the aesthetic abilities of the dental restoration and this progressed to the production of many different types of base metal alloys (Conrad *et al.*, 2007). There was a demand for a strong alloy that could withstand the production processes within the laboratory (Conrad *et al.*, 2007).

Numerous studies have advocated that certain metal oxides were not colour stable after they were subjected to various firing temperatures (Sahin *et al.*, 2010). In conventional porcelain-fused-to-metal restorations, there are numerous available alloys on the South African market and final restoration colour may be influenced by different alloys. The different colours of the metal alloy substructures and the different metal oxides which they produce may influence the colour observed (Vichi *et al.*, 2011).

Although predictable strength and reasonable aesthetics incessantly make conventional metal-ceramic restorations popular, demands for improved aesthetics were the driving force between the developments of dental ceramic materials (Conrad *et al.*, 2007).

McLean achieved a breakthrough in 1967 when he introduced the idea of fabricating a high alumina ceramic core structure (Figure 1) (Schmidseder et *al*., 2000).

Figure 1 : High alumina ceramic core structure under transillumination (Procera®, Nobel Biocare, Sweden)

A joint collaboration saw McLean along with the company Vita develop the Vitadur (1968) and Vitadur N system (1976), which at that point dominated the aesthetic treatment of anterior teeth (Schmidseder *et al.*, 2000). This application was recommended solely for single anterior tooth replacement (McLean, 1976).

The platinum-bonded alumina fixed partial denture was introduced in 1982 in order to reduce the problem of fracture through the connector area of a cast-metal framework for a bridge restoration. The oxidised tin coating provided a mechanism for bonding of porcelain which subsequently saw the traditional cast-metal framework less used. The restorative option for the fabrication of bridges was not feasible and its limited use was for the manufacture of jacket crowns (Rosenblum and Schulman, 1997).

Several approaches have been seen over the last two decades to enhance the ceramic strength with microstructure that substantially differs from that of conventional feldspathic porcelains. Common features include a significant crystalline phase in the glassy matrix. This vastly contributes to their physical, mechanical and ultimately the optical properties of the final product (Vagkopolou *et al.*, 2009). Distribution, nature, particle size and amount of the crystalline phase affect the fracture behaviour of the ceramics. Solids mostly expand in response to heating and contract on cooling which is how a slight mismatch between metal and ceramic is created so that the ceramic would be in a state of compression against the metal and so the ceramo-metal or 'porcelain-fused-to-metal crown' was invented (Owen, 2012). Even though ceramo-metal prosthesis proved to be able to withstand occlusal forces, there was still the disharmony between the ceramo-metal prosthesis and the natural dentition (Stevenson, 2010 and Zhao, 2011). Co-efficient mismatch of thermal expansion amongst the various phases can lead to localized stresses at phase boundaries which may vastly improve the overall toughness (Seghi *et al.*, 1995). Table 1 displays an overview of modern ceramics.

Table 1 : Modern ceramics (Owen, 2012)

Modern Ceramics			
	Glass ceramics	Glass infiltrated ceramics	Pollycrystalline ceramics
Composition	Glass-based systems (mainly silica)	Glass-based systems (mainly silica) with fillers, usually crystalline(typically leucite or more recently, lithium disilicate)	Pollycrystalline solids (alumina and zirconia)
Manufacturer	Ivoclar Vivadent	Vita Zahnfabrik	Dentsply
Strength	High in compressive strength and low in tensile strength	Increased flexural strength	High in compressive and tensile strength
Colour matching/ aesthetics	Very high in translucency. Good aesthetics	Good aesthetics and high translucency	Good aesthetics, high in translucency
Use	Anterior crowns, inlays, onlays	Anterior region due to good aesthetics	Anterior and posterior crowns and bridges, abutments, inlays, onlays
Commercial examples	IPS Empress	Inceram	Cercon

According to Stevenson *et al*. (2010), the brand of all-ceramic core material used may well influence the final restoration colour when layering. They further stated that colour instabilities of the layered veneering ceramic differed by brand, dentin porcelain thickness and shade.

2.3 Zirconia

Zirconia possesses a white, crystalline structure and is an oxidized by-product of Zirconium. The Zirconia powder is pressed until a framework free of voids, flaws and cracks appropriate for CAD-CAM milling is obtained (Celik *et al.*, 2008). Zirconia is a high strength material which offers support to the fragile veneering ceramic. It may be designed for computerized milling techniques (CAD-CAM). This CAD-CAM system enables the core structure to be milled from a pre-sintered block of an oxide ceramic. Zirconia is yttria-stabilized zirconium oxide polycrystals which is made from fine particles of ZrO_2 and 3-5% Y_2O_3 forming a partially stabilized tetragonal arrangement after being heat-treated (Celik *et al.*, 2008 and Vagkopolou *et al.*, 2009). The optical opacity of these materials requires them to be covered with veneering porcelain (Alghazzhawi *et al*., 2012).

The zircon mineral is chemically known as zirconium silicate and contains the chemical element zirconium (metal ore) (Celik *et al.*, 2008). Zirconium is atomic number 40 on the periodic table and is a transitional metal. The zircon mineral exists in three forms namely monoclinic, tetragonal and cubic as first observed by Sir David Brewster in 1821 (Celik *et al.*, 2008).

Due to the lack of silica, obtaining a desired level of translucency can be difficult. In an attempt to manipulate light transmission, and thus the value, various stained cores are now available. Although the character and composition of the veneering layer can aid this process, the core still plays a major role. The colour of the core acts to mimic the underlying dentine (Celik *et al.,* 2008). The use of a zirconia core increases the light transmission and depth of translucency throughout the restoration, however, zirconia based systems use a plain white-coloured core which may limit their aesthetic indications (Raigrodski, 2004; Liu, 2013). Further development regarding the white-coloured core led to the use of a relatively translucent core which at the same time can be coloured in shades that correspond to the Vita-Lumin shade guide (Raigrodski, 2004; Kaya, 2013). This allows the shade of the restoration to develop from its intaglio surface to the outer facet of the veneer and gives the final restoration a more life-like look (Raigrodski, 2004; Liu, 2013). There are different ways of colouring zirconia frameworks. The zirconia material in the green-stage form can be dipped in a colour liquid therefore allowing the colour liquid to infiltrate through the zirconia for uniform colouring. Another method of colouring is by painting the zirconia in the green-stage with a colour liquid before sintering, or colouring additives can be mixed to the zirconia powder before pressing the material to a block before sintering (Denry *et al.*, 2008).

Studies have been conducted on changes in stain colour after firing, and clinically significant colour changes have been reported due to pigment breakdown after the zirconia core is exposed to firing temperatures (Celik *et al.,* 2008). Other studies revealed that the zirconia core colour is unaffected with repeated firings so it is assumed that numerous firings had little effect on the measured colour difference (Celik *et al.*, 2008). However, according to a study conducted by O' Brien cited by Celik *et al.*, (2008) perceivable differences ($\Delta E=1$) were identified between the colour of all-ceramic specimens that were fired 3 and 6 times respectively.

This core veneering complex is subject to various cycles of sintering. The influence that the firing cycles have on the final prosthesis is not widely documented. To meet the high demands of patients and to ensure predictability, the colour of the final product needs to be constant. According to Celik *et al.* (2008), processing techniques, with specific focus on temperature, should not alter colour stability.

The veneering process consist of firing procedures at temperatures between 750^{o} C and 900^{o} C followed by cooling of the ceramic restoration. This process is sometimes repeated up to five times. The effects of this repeated firing on the core ceramic properties, including colour distortion, are not widely researched. Previous studies have indicated that repeated heating of the core materials influence the final colour of the restoration even though the temperatures are much lower than the core materials own sintering temperature (Øilo *et al.,* 2007). However, the colour changes that occur are said to be clinically acceptable (Celik *et al.,* 2008). If the colour of zirconia is affected after numerous firings, the results of this study may assist ceramists in making an informed choice of an all-ceramic system.

2.4 Dental zirconia used for the study

LAVA

The Lava All-Ceramic System was introduced in 2002 by 3M Espe. It utilizes CAD/CAM technology in order to produce a densely sintered, high-strength zirconia framework with 3 mol% yttria partially stabilised zirconia polycrystalline content. The system includes a special scanner (Lava scan) used for scanning and design together with CAD/CAM software technology, a computerized milling machine (CAM) (Lava Form), and a sintering oven (Lava Therm). A contact-free optical process that uses white light triangulation is used to scan tooth preparations and edentulous areas. The entire scanning procedure takes roughly five minutes for a crown preparation and twelve minutes for a 3-unit fixed partial denture (Suttor *et al.,* 2001).

Once scanning of the model is complete, the crown or bridge framework is designed on the computer and subsequently milled from a green blank which is a partially sintered zirconia block. This green blank is chalk-like and softer than a sintered zirconia. Milling time is thus reduced, there is nominal tool wear and does not demand a great load from the milling unit (Suttor *et al.*, 2001). In addition, to compensate for shrinkage during the sintering process (20-25%), the CAM produces an enlarged framework structure. The average milling time for a coping and a 3-unit fixed partial denture substructure is approximately 35 minutes and 75-90 minutes respectively (Suttor *et al.*, 2001). The milled framework is porous and can be coloured in one of seven shades. The colour used is an ionic stainer which infiltrates the zirconia completely and thus ensures uniform colour (Suttor *et al.,* 2001). Sintering takes place in a special automated oven, which is programmed to run for 8 hours (Suttor, 2001). This includes the heating and cooling phases. Yttrium stabilised zirconia is used for the Lava zirconia framework giving it its high strength which is in the region of approximately 1200 MPa (Suttor *et al.*, 2001).

Zirkonzahn

The Zirkonzahn system was originally introduced in 2004 as a manually operated system by Enrico Steger (Vichi *et al.*, 2011). It offers the dental technician simplicity in handling and cost efficiency. The zircon-milling system enables the production of both, simple and high complex constructions (Vichi *et al.*, 2011). The characteristics of the Zirkonzahn system includes a fully automated optical structured-light scanner S600 ARTI with scanning software. The system also has a PC and screen using basic modeling software Zirkonzahn Modellier, twin high speed scanning cameras, extra-large measuring field, different mock-up support plates and scans all types of articulators.

The zirconia used is fired in a sinter furnace that requires a specific program designed for Prettau zirconia material. Prettau zirconia is a fully stabilized material which makes it highly translucent and is said to be ideal for full contour crowns without veneering dental porcelain (Jung *et al.,* 2010). It is thermally and mechanically resistant to temperatures up to 2600^{o}C. The flexural strength of Prettau zirconia lays 10% weaker than regular zirconia, however, this deficit is adequately compensated for by the oversized frame dimension. Thus, the actual flexural strength increases by up to 200% (Jung *et al.,* 2010). Prettau zirconia offers seven blank sizes and thicknesses of 16 mm and 22 mm respectively. The staining liquid is painted onto the surface of the zirconia whilst in the porous green state.

Cercon

This system was initially referred to as a computer aided milling (CAM) system because of the absence of a computer aided design (CAD) component. Once the die was ready, a wax pattern of the coping or pontic with a minimum thickness of 0.4 mm was manufactured (Anusavice, 2003). The system scanned the wax pattern and milled a zirconia structure from pre-sintered zirconia blanks in an enlarged size to compensate for the 20% shrinkage. Cercon zirconia is available in 16 A-D shades and one white shade. The system advanced to having the Cercon brain where the die was scanned by a scanner (Cercon brain), designed and milled by its very own milling unit. The milling time for a crown is approximately 35 minutes and 80 minutes for a 4-unit bridge (Anusavice, 2003).

The coping is then sintered in the Cercon heat furnace at a temperature of 1,350°C. The sintering process lasts between 6 to 8 hours. Milling presintered blanks reduces milling time and increases the service lifespan of the unit and instruments (Anusavice, 2003). This ultimately saves on cost in the long run as presintered zirconia is soft and easily milled.

2.5 How does colour perception work?

The integration of several critical factors is required to obtain a successful aesthetic restoration (Vichi *et al.,* 2011). This includes the source of light used to evaluate colour, one's perception of colour, the surface and structural features of both teeth and the restorative materials, and knowledge of basic principles of colour perception. Dental personnel must understand light, colour and related characteristics of porcelain (Vichi *et al.*, 2011).

Light can be described as electromagnetic radiation that can be perceived by the human eye (Anusavice, 2003). The eye is sensitive to wavelengths from approximately 400nm (violet) to 700nm (dark red) as shown in Figure 2.

The appearance properties (hue, value, chroma) are determined by the reflected light intensity and the joint intensities of the wavelengths existing in incident and reflected light. For an object to be visible, light incident must be reflected or transmitted on it from an external source. The incident light is usually a combination of numerous wavelengths known as polychromatic (Vichi *et al.*, 2011; Sarafianou *et al.*, 2012). Incident light is selectively scattered, absorbed or both by certain wavelengths. Although certain wavelengths are reduced in magnitude, the spectral distribution of the transmitted or reflected light resembles that of the incident light (Anusavice, 2003; Sarafianou *et al.*, 2012). The phenomenon of vision can be demonstrated by considering the human eye response to light reflected from an object. A light from an object that is incident on the eye is focused in the retina. It is then converted into nerve impulses that are transmitted to the brain (Burkinshaw *et al.,* 2004). Cone-shaped cells in the retina are responsible for colour vision (Vichi *et al.*, 2011). These cells have a threshold intensity essential for colour vision. They also display a response curve related to the wavelength of the incident light. Figure 2 illustrates such curves for individuals with colour-deficient vision and for individuals with normal colour vision.

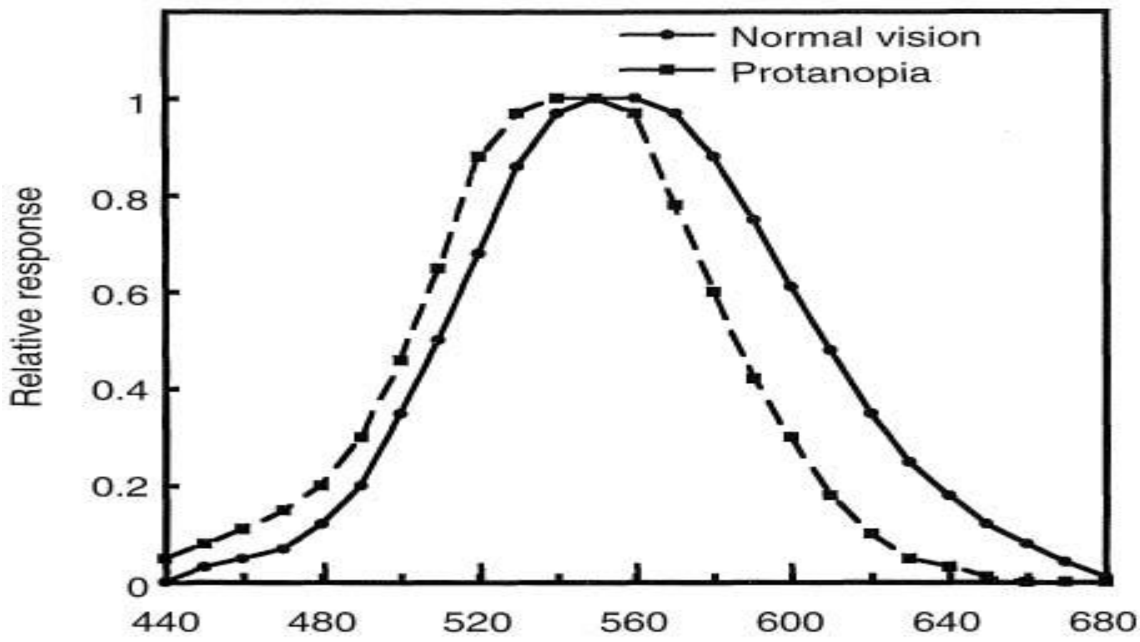


Figure 2 : Relative visual response of humans to wavelength of light for a normal observer and one with protanopia (reti-green) colour blindness (Anusavice, 2003).

Protanopia is experienced by 1% of the male population and 0.02% of the female population. Figure 2 shows the normal observer curve. This indicates the human visual responsiveness to light reflected or emitted from a particular source or object. This figure indicates that the eye is least sensitive to light at the red or blue regions and most sensitive at the green-yellow region (wavelength of 550nm) (Geary and Kinirons, 1999).

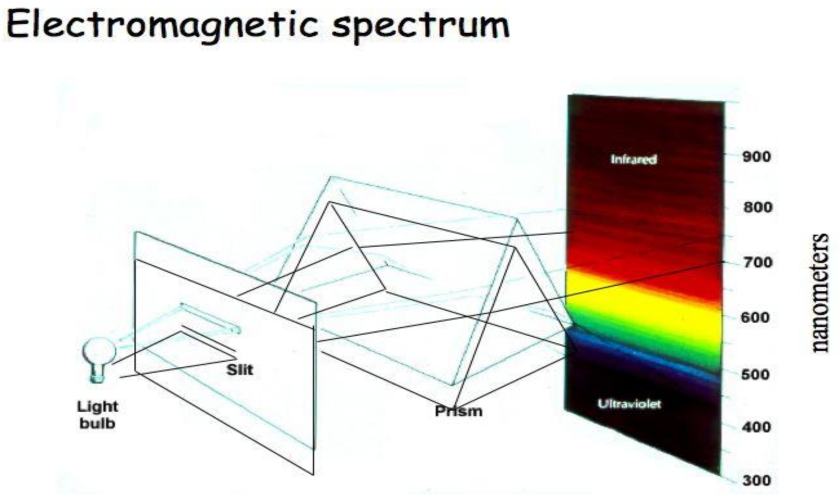


Figure 3 : Spectrum of visible light ranging in wavelength from 400 nm (violet) to 700 nm (red) (Anusavice, 2003)

The most visually perceptible region of the equal energy spectrum under daylight conditions is between wavelengths of 540 and 570 nm with a maximum visual perceptibility of 555nm (Anusavice, 2003) as seen in Figure 3**.** A decrease in the eye's response as well as colour fatigue may result due to constant stimulation by a single colour. The signals received from the retina are processed by the brain in order to produce the psychophysiological perception of colour. Different types of colour blindness are consequences of defects in certain portions of the colour sensing receptors. Thus, human observers vastly differ in their ability to distinguish between colours (Celik *et al.*, 2008). While the colorimeter is more precise than the human eye in establishing minute colour differences in coloured objects, it can be extremely inaccurate when used on rough or curved surfaces. The human eye is better able to differentiate between two colours seen on smooth or irregular surfaces (Celik *et al.*, 2008).

The production of colour perception with a pigment is a substantially different phenomenon from that achieved by optical refraction, reflection and dispersion. The colour of a pigment is established by selective absorption and selective reflection (Celik *et al.*, 2008). An example of this is when white light is reflected from a red surface, the light with a different wavelength to that of red is absorbed and only the red light is reflected (Celik *et al.*, 2008). Hence, if a red hue is present in a ceramic crown and red wavelength is absent in the light beam, the tooth will appear as a different shade (Anusavice, 2003). If the restoration surface or tooth itself is rough, most of the light will be scattered and little will penetrate the structure. In certain instances, almost no colour can be seen (Anusavice, 2003).

The appearance of an object is greatly influenced by the geometric characteristics of appearance such as transparency, translucency, gloss, opacity and optical occurrences such as opalescence, metamerism and fluorescence (Vichi *et al.*, 2011). Whilst these diverse aspects affect colour perception of a tooth, the optical characteristics of a tooth were considered quite complex and finding a proper aesthetic result was considered more of an art form rather than a standardized and predictable procedure (Vichi, *et al.,* 2011).

2.6 Dental colour matching instruments and systems

History

In the past, several authors tried to objectively quantify tooth colour by either one's own visual shade selection and shade guides that exhibited significant shortcomings (Chu *et al.*, 2010). This was done by assessing the quality and quantity of light required to properly analyse a shade through the association between extracted teeth and the shade guides or the development of the early shade guides and shade measuring instruments (Chu *et al.*, 2010).

A new industry in dentistry occurred in the late 1990's. It was the commercially available instrument-based colour measurement systems, with the advancement of the ShadeScan system (Cortex Machina, Montreal, Canada) (Chu *et al.*, 2010). This was the paramount effort toward a shade analysis system for comprehensive tooth surface measurement and the advancements occurred rapidly thereafter.

To date, shade-matching technology had been established in order to increase the success of communication, verification, colour matching and reproduction in clinical dentistry (Yamamoto *et al.*, 2012). Ultimately, the primary aim was to increase the efficiency of aesthetic restorative work within any practice (Chu *et al.*, 2010).

Shade guides

Visually using a dental shade guide is the most commonly used method of tooth shade matching. In the year 1956, Vita introduced the first shade guide for the measurement of the colour of ceramic systems (Yamamoto *et al.*, 2012). Although not entirely perfect, it introduced some visual parameters that are still used by dental personnel today (Yamamoto *et al.*, 2012). The Vitapan Classical Shade guide consists of 16 tabs. These tabs are arranged into four groups which is based on hue and within the groups according to increasing chroma (Paravina, 2002). The Ivoclar-Vivadent Chromascop is another popular shade guide. The Chromascop is arranged in groups based on the hue (1=white, 2=light yellow, 3=dark yellow, 4=grey and 5=brown) and within the groups according to increasing chroma (from 10 – 40).

The late 1990’s introduced the L*a*b* system and one of the first clinical results was the development of the Vitapan 3D Master shadeguide. This colour system consists of eleven sets of tooth shaped fired porcelain samples built up with cervical, dentinal and incisal powders.

Figure 4 : Vita 3D master Shade Guide

These eleven sets consists of twenty six samples ranging from lowest to highest intensity, from lightest to darkest value and from yellow to red (Paravina, 2008). The Vita 3D master shade guide (Figure 4) is based on a colour classification principle in which three dimensions of colour, value (brightness), chroma (intensity of colour) and hue (the colour itself) are considered equally so that shade determination can be easily carried out (Paravina, 2008 and Corciolani, 2009). This shade guide addresses the most imperative element of tooth shade selection: a scientific colour distribution with a systematic arrangement of shades within the natural tooth colour space (Hammad, 2003).

Electronic shade taking devices

Spectrophotometers, colorimeters and imaging systems are instruments for measuring light. An intra-oral measuring device has been designed to fit the needs of clinical dentistry. It obtains data such as tooth translucency or statistics associated with colour communication, reproduction and verification on the corresponding shade tabs (Chu *et al*., 2010). These aspects including price limitation have dictated the dental market with less emphasis being placed on scientific aspects, such as providing colour formulation or reflectance values, less emphasized (Chu *et al*., 2010). Optical properties of human teeth significantly add to non-dental applications. This is due to the fact that they are curved, small, translucent, multilayered and display colour changes in all directions (gingival to incisal, mesial to distal and labial to lingual) (Hammad, 2003). Hence, the accurate repositioning (measurement of the same area) is of critical importance for both clinical and research use of dental colour matching devices (Chu *et al*., 2010).

a) Colorimeter

The colorimeter is a comparatively simple and low cost instrument designed to measure colour on the basis of three axes by using a filter that stimulates the eye. Colorimeters are relatively cheap, portable and have a small opening for reading measurements. However, their accuracy is questionable and limitations are vast which led to the failure of the chromascan system (Chu *et al*., 2010).

b) Spectrophotometer

A spectrophotometer is a more sophisticated device that delivers illuminating light. The material that is measured is infiltrated by this light, travels through the material, and is either re-emitted or absorbed. Some of the photons that are re-emitted follow pathways which lead outside the window area. This results in edge loss (Spyropoulou *et al.*, 2011). Spectrophotometric measurement benefits include the ability to analyze the main components of a series of spectra and the capacity to convert spectrophotometric measures to numerous colour measures. Even though the initial spectrophotometers were accurate, they were expensive, bulky, difficult to manage and not easy to accurately calibrate (Spyropoulou *et al.*, 2011).

c) Imaging

Advances in the dental industry have resulted in sophisticated charge-coupled devices (CCD's), which sparked the development of electronic devices that are said to accurately measure the colour of teeth (Table 2). These devices range from software that can be utilised in combination with images taken with a digital camera to 'pure' spectrophotometers and 'pure' colorimeters to very latest innovation of digital images that are combined with the spectrophotometer or colorimeter (Chu *et al.*, 2010).

Table 2: Instruments and Software for colour matching in Dentistry (Chu *et al.*, 2010)

Product	**Manufacturer**	**Device type**	**Measurement area**	**Relative cost**
ClearMatch	Clarity Dental, Salt Lake City, UT	Software, digital image analysis	Complete tooth image	Low
Easyshade Compact	Vident, Brea, CA	Spectrophotometer	5-mm probe diameter	Low
ShadeVision	X-rite Grandville, MI	Imaging colorimeter	Complete tooth image	Moderate
CrystalEye	Olympus America, Center Valley, PA	Imaging Spectrophotometer	Complete tooth image	High
SpectrShade Micro	MHT, Niederhasli, Switzerland	Imaging Spectrophotometer	Complete tooth image	Moderate
Shade-X	X-rite Grandville, MI	Spectrophotometer	3-mm probe Diameter	Low

2.7 *Colour science*

Conventionally the colour of teeth has been described in terms of Munsell colour parameters namely: hue, value, chroma (Anusavice, 2003). However, in order to aid the quantification of colour differences, L*a*b* colour system was used (Anusavice, 2003; Bona *et al.*, 2009). The three coordinates to define colour are L*, a* and b* as seen in Figure 5. The L* coordinate describes the level of greyness of a colour along the black-white axis. The greater the L* value is, the lighter the specimen. The a* coordinate describes the colour in reference to the red-green axis which is a measurement of chroma. A negative a* relates to the greenness of a specimen and a positive a* the redness. The b* coordinate relates to the chroma along the yellow-blue axis. A positive b* relates to the amount of yellowness, whilst a negative b* relates to the amount of blueness of the specimen (Celik *et al.*, 2008; Bona *et al*., 2009). This colour system was developed by the International Commission on illumination. Delta L (ΔL*), Δa*, Δb* are the difference in the colour space parameters (Figure 5).

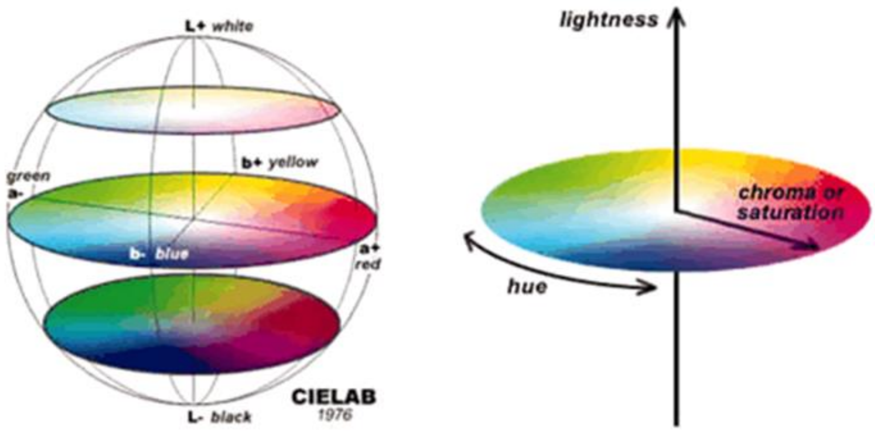


Figure 5 : Munsells colour system (Anusavice, 2003)

Delta E Values are used to establish whether the changes in the overall shade can be perceived by the human eye. It has been noted that colour differences within the 1 to 2 ΔE range, can be correctly judged by human observers (Sahin *et al.,* 2010). A ΔE value of 1.6 was deemed a colour difference that could not be detected by the human eye. Sahin *et al.* (2010) states that when the ΔE values are greater than 2 ΔE units, human observers can detect colour difference between two colours. Due to the fact that it is difficult to light conditions in the oral cavity, the colour difference value limit of 3.7 ΔE units is said to be clinically acceptable as confirmed by Johnston and Kao (1989). These authors also suggested an extended visual rating scale (EVRSAM) to further understand the clinical significance of the numerical results of instrumental analysis evaluation of dental material colour (Table 3).

Table 3. Extended visual rating scale (EVRSAM) Johnston and Kao, (1989)

ΔE	Clinical significance
0	Excellent aesthetics with accurate colour choice, not being clinically perceived, or only with great difficulty.
2	Very slight difference in colour, with very good aesthetics.
4	Obvious difference, but with an average acceptable to most patients.
6	Poor aesthetics, but within the limits of acceptability.
8	Aesthetics are very poor and unacceptable to most patients.
10	Aesthetics are totally unacceptable.

The Spectroshade system calculates the three colorimetric coordinates of the tooth (L - Lightness, C - Chroma, h - Hue) and of the shade guide which are also L -Lightness, C - Chroma, h – Hue (Johnston and Kao, 1989).

It is then possible to calculate the colour differences that are:

- ΔE - the overall colour difference that establishes the colour variance between the tooth and shade guide on the 3-dimensional colorimetric space.

- ΔL - is the difference in lightness, excluding chroma and hue, less influent in human eye perception (Chu *et al.*, 2010).

2.8 *Factors affecting the colour of ceramics*

The main reason for the choice of ceramic as a restorative material is its ability in matching adjacent tooth structure in terms of translucency, colour and chroma. The structure of a tooth influences its colour. Dentin is more opaque than enamel and acts to reflect light. Enamel is a crystalline structure and is the layer over dentin which is made of tiny prisms or rods cemented together by an organic substance (Anusavise, 2003).

The cementing substance and the indices of refraction of the rods are different (Anusavice, 2003). This results in a light ray being scattered by reflection and refraction thus producing a translucent effect and sensation of depth as the light ray reaches the eye (Anusavise, 2003; Spink *et al.*, 2013). The light ray that strikes the tooth surface is partly reflected and the remainder is scattered by the enamel (Anusavise, 2003; Spink *et al.*, 2013). If dentin is not present as in the tip of an incisor, some of the light may be absorbed and transmitted within the oral cavity. This may result in the area appearing more translucent than the gingival area. Thus, due to the fact that the law of energy conservation must apply, the following relationship displays the five energy components making up energy (E) of the incident light (Anusavice, 2003).

Incident = scattered + reflected + absorbed + transmitted + fluoresced

While some of the absorbed light may be converted into heat, some may be transmitted to the eye as fluorescent energy. When the ultraviolet rays of daylight contact teeth, some of the radiant energy is transformed into light of one or more colours such as yellow, red and orange. Light rays can also be spread, giving a colour or shade that varies in different teeth and types of light. This phenomenon is better known as metamerism (Burkinshaw, 2004).

It is impossible to imitate such an optical system perfectly, however, the laboratory technician can reproduce aesthetic characteristics to the extent whereby the difference is conspicuous only to the trained eye (Anusavice, 2003). Dental porcelains are pigmented by the inclusion of oxides to obtain the desired shade. This is done by the dentist using a shadeguide, however, there are variances in the different types. Not only are the shadeguide tabs much thicker than ceramic used to build up a crown, they are also more translucent than teeth and crowns that are backed by non-translucent dentin core. Most of the incident light is transmitted through a tab whereas on a tooth much of it is reflected back except at the incisal edge (Burkinshaw, 2004). The production of colour sensation with a pigment is a physically different phenomenon from that obtained by optical dispersion, reflection and refraction (Anusavice, 2003). Selective absorption and selective reflection determines the colour of the pigment. An example of this is when white light is reflected from a red surface, all the light with the wavelength different from that of red is absorbed and only the red light is reflected (Anusavice, 2003). This ultimately means that if a red hue is present in a ceramic crown, but the red wavelength is absent in the light beam, the tooth will appear as a different shade (Anusavice, 2003).

Metamerism

Two types of metamerism exist: Illuminant and observer.

a) Illuminant metamerism

Illuminant metamerism is referred to a situation during which two coloured samples, when viewed simultaneously, match under one set of lighting conditions but differ under another. An example may be two objects that appear to be the same colour when viewed under daylight but display distinctive colour differences when viewed under tungsten light (Burkinshaw, 2004).

b) Observer metamerism

However, observer metamerism arises because of the variances that may occur in the colour vision of the observers, angle of view and area of view (Burkinshaw, 2004). The dental technician should, therefore, take steps to reduce metamerism. The two crucial steps in obtaining the desired result are obtaining a colour that would harmoniously integrate itself with the surrounding biological tissue and reproducing this colour in the final prosthesis (Burkinshaw, 2004). This may be achieved from the initial stage by obtaining the correct shade of the zirconia core.

Colour constancy

Colour constancy is associated with a distinct coloured object as opposed to metamerism which refers to a pair of coloured objects. This phenomenon is displayed by the majority of natural objects: they appear to have the same colour even when viewed under different light sources (Burkinshaw, 2004).

Optical Properties

Optical properties is defined as one of the effects of a substance or medium on light or other electromagnetic radiation passing through it, such as refraction, scattering, polarization and absorption (Bona *et al.*, 2009). Numerous factors such as translucency, opalescence, fluorescence, shape properties, surface texture, porcelain brand and batches, condensation technique and the number of porcelain firings may have an effect on the final shade of porcelain (Celik *et al*, 2008). Absorption of a specific wavelength is related to shading. This leads to a lower translucency of the material which is an important step in realizing a high translucency despite colouring. Translucency is influenced by the thickness of the material. A greater thickness leads to higher absorption and consequently higher opacity (Behrens, 2008). Overall aesthetic appearance of a restoration should match the oral environment. Therefore, the optical properties of the restorative material need to be similar to that of natural teeth. The shade and colour of a crown is influenced by:

1) The chemistry and absorption spectra of the object (Behrens, 2008).
2) The kind of light falling onto the object (Behrens, 2008).
3) The sensory impression of the eye of the observer (Behrens, 2008).

Light transmission is permitted with all ceramic restorations, thereby improving the colour and translucency of the restoration. Dental laboratory technicians are allowed to fabricate an aesthetic restoration with individual character due to the strength of the zirconia coping and layering techniques of the veneering material (Celik *et al.*, 2008).

Opalescence

Opalescence is a term given to substances exhibiting similar properties to opal stone when subjected to reflected or transmitted light (Raptis *et al.*, 2006). Natural teeth are able to fluoresce under UV light thus making teeth look whiter and brighter (Behrens, 2008). This phenomenon is created with various proteins and the inorganic matrix of natural teeth (Raptis *et al*., 2006).

The complex nature of light as it interacts in refraction, reflection and absorption of microscopic material differences results in metameric differences in colour that warrants continual updated evaluation especially for aesthetic dental materials (Behrens, 2008).

2.9 Effects of sintering on grain size and translucency

The amount of light absorbed, transmitted and reflected depends on the amount of chemical and crystal components inside the core materials and the particle size compared with the incident light wavelength (Jiang *et al*., 2011). An increase in the translucency of the zirconia core can vastly improve the aesthetic characteristics of dental restorations (Jiang *et al*., 2011). Essentially, light scattering determines the translucency of the zirconia ceramic. Low porosity is required for increased translucency.

Thickness is a co-variable relative to translucency (Spyropoulou *et al.,* 2011). Factors influencing translucency of zirconia include:

- Porosity
- Thickness
- Sintering temperature and atmospheric conditions
- Primary particle size/grain size
- Additives

Amongst these factors, the sintering temperature has a major impact on the densification processes and microstructure of Y-TZP nanopowders (Jiang *et al*., 2011). A study conducted by Ozturk *et al.* (2008), tested change in colour and translucency of DC-Zirkon ceramic cores of different thicknesses. The resultant ΔE values between 3 and 5 firings for DC-Zirkon all-ceramic specimens with 0.5mm thicknesses were the highest and decreased with subsequent firings. Furthermore, as the thickness increased, ΔE values increased as well. Heffernan *et al.* (2002), reported an opacity of 1.00 (which is completely opaque) for a 0.5mm thick In-Ceram zirconia core, however, In-Ceram zirconia which is glass infiltrated is structurally different from yttria stabilised pollycrstalline zirconia. The same opacity value was reported by Chen

(2008), for Cercon cores at 0.5mm thickness. Baldissara *et al.,* 2010 further reported Cercon to be highly opaque in a study which included Lava frames of 0.3mm and 0.5mm thickness which were found to be the most translucent material amongst those that were tested.

A study conducted by Kim *et al.,* 2013 stated that grain size and translucency of zirconia changed according to changes in sintering conditions. The translucency of dental ceramics can be calculated in three ways according to Kim *et al.* (2013),: direct transmission, total transmission, and indirect measurements. Light scattering relies on the grain size and wavelength of incident light (Kim *et al.*, 2013). According to Kim *et al.*, 2013, when grain size and wavelength of incident light are in a comparable range, the amount of light scattering decreases the light transmittance. When grain size is larger than the wavelength of incident light, the amount of light scattering becomes independent of the wavelength of incident light and inversely proportional to the grain size. Kim *et al.* (2013), also stated that light transmittance decreases with an increase in grain size. They also concluded that the grain size of dental zirconia is affected by sintering conditions (time). In essence, the shorter the sintering time, the smaller the size of the grain and an increase of the light transmittance values of the final zirconia restoration.

2.10 How does colourization influence translucency?

With various zirconia core shades available on the market, it is beneficial to evaluate the effect of shaded zirconia on the translucency of ceramic crowns. A study conducted by Spyropoulou *et al.* (2011), on the translucency of three Procera zirconia shaded core materials revealed that there is a significant difference in translucency between the light and intense shades and the medium and intense shades. The light and medium shades displayed no significant difference. Jiang *et al.* (2011) and Anusavice (2003) noticed that colourants of ceramic could not affect the crystallization process to control the mean grain size for the translucency.

CHAPTER THREE

RESEARCH METHODOLOGY

3.1 Study design

This study was designed to determine which zirconia system best maintains colour stability after repeated firing. The zirconia material used was selected based on their properties as well as the willingness of the manufacturer to support the study. The systems selected for this study were Lava (3M ESPE, USA), Zirkon Zahn and Cercon (Dentsply DeguDent, USA). These CAD-CAM systems are readily available in South Africa and are also popular abroad. Establishing which brands were commonly used ensured that the results will have applicational value in the dental industry. The zirconia based ceramics used in the study consist of pure, partially stabilised zirconia particles (95% ZrO2, stabilised by 5% Y2O3) with a mean grain size of 0.4 µm. The experimental (quantitative and comparative) method was used.

The aim of this study was to determine the effects of three firings on the shade of three different zirconia systems available in South Africa. Each sample group was obtained directly from the manufacturer. Green state zirconia blocks from Cercon (Dentsply DeguDent, USA), Lava (3M ESPE, USA), and Zirkon Zahn (Zirkonzahn Gmbh, Bruneck, Italy) were trimmed, finished and sintered to a final thickness of 0.5mm according to the manufacturers' instructions. A sample size of eighteen was used for each experimental group. In each group, nine specimens were shaded and nine were unshaded. All specimens were subjected to spectrophotometer testing after each firing and SEM analysis after one and three firings to determine microstructural changes that could be present in the different sample groups. There was a total of 54 specimens x 3 firings; n = 108.

The initial phase was to fabricate eighteen specimens for each of the zirconia groups. Nine specimens in each sample group were shaded using the A2 shade and nine were unshaded. The shade from the "A" group was selected due to the fact that this group accounts for at least 65% of clinical shade selections (Celik *et al*., 2008). Each specimen was encoded to ensure that this research remained a blind study. Thereafter, each specimen was tested for colour accuracy using a spectrophotometer (Spectroshade Micro MHT, Niederhasli, Switzerland). Once the results were documented, each sample group was exposed to one, two and three firings, respectively, and had their shade taken and recorded. The recorded data from the strophotometer were analysed with the assistance of a statistician.

Once spectrophotometer testing was complete, one shaded and one unshaded specimen from each sample group was sent for SEM analysis. The selected copings were broken in half in order to obtain a cross-sectional view of the specimen. The data was then decoded and analysed.

3.2 Limitations

- The shade was tested on core material in the absence of veneering material and may not be representative of clinical conditions. The absence of veneering material would result in greater accuracy of core shade after firing (Stevenson, 2010).

3.3 Specimen Preparation

Zirconia copings

A maxillary left central incisor shape of a resin model tooth was used as the original abutment tooth (Figure 6). After a master die was formed by adjusting the model tooth on a pattern resin (DuraLay, Reliance Dental Manufacturing Company, Worth, IL), it was used as an impression template. Individual trays were prepared and used to make the impression using Impregum impression material (3MEspe, St. Paul, MN, USA) and thereafter poured in die stone in order for the die to be detected by the respective scanners of the Lava, Zirkon Zahn and Cercon systems, respectively.

Figure 6: Maxillary left central incisor used as original abutment

The stone die was mounted into the respective scanning unit linked to a computer. The scanners mapped the surface of the die and obtained a digital prescription which had a uniform thickness of 0.5mm each. These digital prescriptions were transmitted electronically to the respective milling units, milled, stained and sintered at their specified temperatures.

For uniformity, the thickness of each specimen was 0.5 mm (after the sintering procedure). In accordance to manufacturer's guidelines all preparation, manufacturing and sintering of each zirconia ceramic system was performed by the same hand (Pecho *et al.*, 2012). The specimens were fired in a vacuum furnace (Vita dentine firing program, Vita vacumat 30) (temperature increase rate: 30^0C for one minute; holding temperature: 930^0C for one minute).

The sample

The sample groups consisted of 18 Lava, 18 Cercon and 18 Zirkon Zahn zirconia copings (Table 4). The control and experimental groups consisted of 18 specimens each respectively (Table 4), with 9 specimens in each group shaded using an A2 shade and the remaining being left unshaded.

Table 4. The summarised sample composition

			Group		Total
			Control	Experimental	
Sample Group Name	CERCON	Count	18	18	36
		% within Group	33.3%	33.3%	33.3%
	ZIRKON ZAHN	Count	18	18	36
		% within Group	33.3%	33.3%	33.3%
	LAVA	Count	18	18	36
		% within Group	33.3%	33.3%	33.3%
Total		Count	54	54	108
		% within Group	100.0%	100.0%	100.0%

Spectrophotometric testing

The colour of each specimen was obtained using the spectrophotometer (SpectroShade Micro MHT, Niederhasli, Switzerland) which combines digital colour imaging with a spectrophotometer (Figure 7). It is one of the most user friendly of the various shade taking dental instruments.

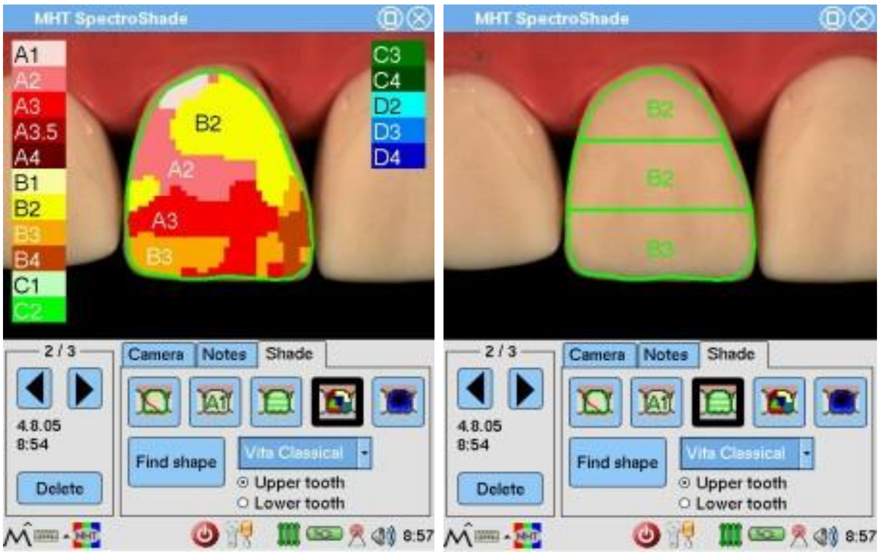


Figure 7: Actual images of specimen on Spectroshade (SpectroShade Micro MHT, Niederhasli, Switzerland)

This device captures the images with a $45/45^0$ geometry and a polarized filter is used to circumvent specular reflection from the surface of the tooth which can influence colour assessment (Vichi *et al.*, 2011). The differentiated images are then used for calculation and colour analysis as well as for comparison with a data set of several shade guides stored in the instrument. Its software can perform a number of functions including both fine and coarse colour shade mapping (Vichi *et al.*, 2011).

Calibration

Prior to any testing being done, the spectrophotometer was switched on for approximately thirty minutes before calibration as per manufacture recommendations.

The Spectroshade was calibrated after each sample group to ensure that the settings were exactly the same and that no calibration differences had occurred. The calibration programme was selected and the Spectroshade was calibrated on the white tile first and then the green tile. The shade was thereafter taken.

The system automatically recognizes if the measurement belongs to an upper or a lower tooth. The system was checked to ensure that this recognition worked correctly.

Coding of zirconia specimens for identification

As seen in Figure 8:

a1, b1, c1 - Groups exposed to one firing cycle

a2, b2, c2 - Groups exposed to two firing cycles

a3, b3, c3 - Groups exposed to three firing cycles

Figure 8 : Sample groups were named a, b and c

Each specimen was marked with one, two and three dots respectively

The dots indicated:

o - one firing

o o - two firings

o o o - three firings

Each specimen had its shade taken prior to firing and this was used as the control to compare each group after the respective firing cycle. Best match using ΔE: the system finds the closest shade guide tab to the tooth, taking into consideration all three components of a colour (recommended). Best match using ΔL: the system finds the closest shade guide tab to the tooth, taking into consideration only the lightness of the colour (Sahin *et al.*, 2010).

All specimens were exposed to the first firing. The zirconia was placed on the fibrous pad (Figure 9) (Vita Zahnfabrik, Bad Säckingen, Germany) with the labial side facing upwards as this was the side that the shade was to be taken.

The programme setting for each firing was standard for each group. The Vita Zahnfabrik, (Germany) firing furnace was used in this study, which:

- Has proper calibration during the firing of the ceramic restorations.
- Has a precise and reliable thermostat.
- Has software that allowed for accurate monitoring of each cycle.
- Has programmable memory recalled cycle settings

- Has a temperature setting of each firing cycle for the different zirconia systems precisely the same.

Table 5 describes the stages of the sintering cycle. Each specimen was handled in the same way.

Table 5: Programme and its setting

Preheating time	Four minutes
Rate of climb	30^{0}C per minute
End temperature	930^{0}C
Holding temperature	One minute, reduce vacuum, open slightly
Slow cool	Up to 600^{0}C

Figure 9: Uniform baking of specimens on fibrous pad

After the first firing, all specimens were placed in their respective groups.

Group a1, b1 and c1 were isolated and had their shade taken after the first firing to determine if there are any colour changes at this stage.

The zirconia copings were placed in the same position in the putty of the mouth simulator each time to ensure that the results were uniform. Each specimen had adjacent teeth (Fig 10). A glaze liquid was applied to each coping so as to simulate the moist oral environment.

Fig 10 : Mouth simulator with specimen mounted between two adjacent teeth

Groups a2, b2, c2, a3, b3, c3 were then exposed to their second and third firing cycles respectively. The same protocol was followed to minimize influence of the technique on results.

3.4 Scanning electron microscope analysis

Only one specimen from the three groups before firing and after the third firing was selected to undergo analysis. A scanning electron microscope was employed to examine the fractured surfaces of the yttrium stabilized tetragonal polycrystalline zirconia. The specimens were mounted onto 0.5” aluminium stubs for scanning electron microscope (Figure 12). The specimens were prepared for imaging by inserting the stubs into the scanning chamber. They were then analysed by scanning electron microscopy in combination with the software.

Figure 11: Stubs on which specimens were mounted for SEM analysis

3.5 Statistical Analysis

The raw data for the statistical analysis was obtained from the spectrophotometer readings recorded before and after the firings. The readings were captured on an excel spreadsheet and analysed using SPSS version 22.0.

The results chapter presents the descriptive statistics in the form of graphs, cross tabulations and figures. The data were compared among the three groups using Pearson's chi square tests where the data were measured as a binary or nominal variable, and using t-tests where a measured normally distributed variable was compared. The general linear model (GLM) was used to analyse combinations of the variables and their effects on the dependant variable.

CHAPTER FOUR

RESULTS

Statement of findings, interpretation and discussion of the primary data

4.1 Introduction

The results and findings obtained from this study are presented and discussed in this chapter.

4.2 Results of the sample composition

Table 6 and Figure 12 provide a summary of the results for the sample composition. It is observed that all the unshaded specimens had a result type of B1. The Cercon results for both the control and experimental groups reveal the same result; B2 shade is maintained before and after the third firing. This suggests that Cercon is a stable material. It is important to note that all copings were received directly from the manufacturer and variables such as the type of pigment used and duration which the copings were placed in the staining liquid all contribute to the final product. The Zirkon Zahn experimental shaded specimens had variations in the results. All 9 specimens in the control group resulted in A2 shade after spectrophotometer testing. However, the experimental group resulted in 5 *shaded* B2, 3 *shaded* A2 and one *shaded* A1 after 3 firings. This suggests that Cercon maintains better colour stability than Zirkon Zahn after repeated firing. The Lava *shaded* specimens for both control and experimental groups had variations in the results, there were 4 *shaded* A1 and 5 shaded B2, however, both the *unshaded* and *shaded* in the control and experimental groups kept the same colour. These results were deduced as 9 *unshaded* in the control group and 9 *unshaded* in the experimental group, 4 *shaded* A1 in the control group and 4 *shaded* A1 in the experimental group and 5 *shaded* B2 in the control group and 5 *shaded* B2 in the experimental group. The experimental group in Table 6 refers to the third firing.

Table 6. Summary of the results for the sample composition

Sample Group Name			Colour	A1 Count	A1 Percent	A2 Count	A2 Percent	B1 Count	B1 Percent	B2 Count	B2 Percent
Sample Group Name	CERCON	Control	Unshaded	0	0.0	0	0.0	9	8.3	0	0.0
			Shaded	0	0.0	0	0.0	0	0.0	9	8.3
		Experimental	Unshaded	0	0.0	0	0.0	9	8.3	0	0.0
			Shaded	0	0.0	0	0.0	0	0.0	9	8.3
	ZIRKON ZAHN	Control	Unshaded	0	0.0	0	0.0	9	8.3	0	0.0
			Shaded	0	0.0	9	8.3	0	0.0	0	0.0
		Experimental	Unshaded	0	0.0	0	0.0	9	8.3	0	0.0
			Shaded	1	0.9	3	2.8	0	0.0	5	4.6
	LAVA	Control	Unshaded	0	0.0	0	0.0	9	8.3	0	0.0
			Shaded	4	3.7	0	0.0	0	0.0	5	4.6
		Experimental	Unshaded	0	0.0	0	0.0	9	8.3	0	0.0
			Shaded	4	3.7	0	0.0	0	0.0	5	4.6

A plot of the results revealed the following:

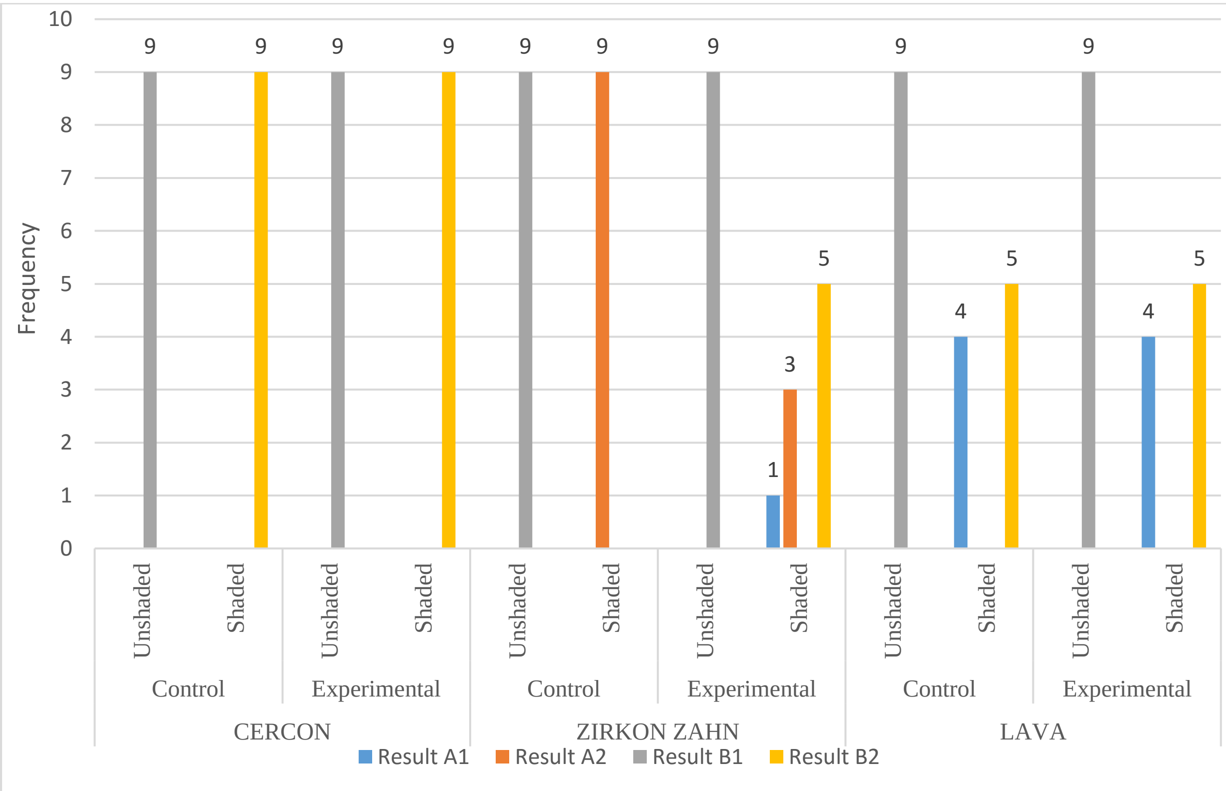


Figure 12: Graph illustrating the results of the sample composition

4.3 The distribution pattern between results after third firing and type of shading

The Pearson's chi-square test was conducted in order to determine whether there was a relationship between results after the third firing and type of shading per group (Table 7). In all instances, the chi-square p- values were less than the value of significance of 0.05. This implies that there was a significant relationship between results of shade after the third firing and the type of shading. That is, the type of shading did play a role in terms of the results achieved. In all sample group names, *p* = 0.000.

Table 7: Influence of shade on results

						Result
Sample Group Name	CERCON	Group	Control	Shaded / Unshaded	Chi-square	18.000
					Df	1
					Sig.	0.000*
			Experimental	Shaded / Unshaded	Chi-square	18.000
					Df	1
					Sig.	0.000*
	ZIRKON ZAHN	Group	Control	Shaded / Unshaded	Chi-square	18.000
					Df	1
					Sig.	0.000*
			Experimental	Shaded / Unshaded	Chi-square	18.000
					Df	3
					Sig.	0.000*
	LAVA	Group	Control	Shaded / Unshaded	Chi-square	18.000
					Df	2
					Sig.	0.000*
			Experimental	Shaded / Unshaded	Chi-square	18.000
					Df	2
					Sig.	0.000*

Sig - significance

4.4 Results based on the one-way MANOVA analysis

SPSS produced many different tables in its one-way MANOVA analysis. In this section, tables are presented to show the results from the one-way MANOVA and Tukey post-hoc tests. The results for ΔE (change in colour) and ΔL (change in lightness) were compared for the various variables that constituted the sample. As multiple readings were done on each sample, the GLM procedure was used. The general linear model is the procedure on which the GLM Multivariate is based, whereby factors and covariates are assumed to have linear relationships to the dependent variables.

The Multivariate tests table indicates the actual result of the one-way MANOVA. These variables are used in conjunction with the Wilks' Lambda row (Table 8). Wilks' Lambda tests are used to test whether there are differences between the means of identified groups on a combination of dependant variables. To determine whether the one-way MANOVA was statistically significant one needs to look at the 'Significance' ("sig") column (Table 8). It is observed from the table that all "Sig" values are less than the level of significance of 0.05, whilst others are not. Therefore, we can conclude that for all *p*-values < 0.05, there was significant dependence on the effect variable.

Table 8. Actual result of the one-way MANOVA

	Value	F	Hypothesis df	Error df	Sig.	Noncent. Parameter	Observed Power[b]
Pillai's trace	0.277	11.660[a]	2.000	61.000	0.000	23.320	0.992
Wilks' lambda	0.723	11.660[a]	2.000	61.000	0.000	23.320	0.992
Hotelling's trace	0.382	11.660[a]	2.000	61.000	0.000	23.320	0.992
Roy's largest root	0.382	11.660[a]	2.000	61.000	0.000	23.320	0.992

Each F tests the multivariate effect of Group. These tests are based on the linearly independent pairwise comparisons among the estimated marginal means.

a. Exact statistic

b. Computed using alpha = 0.05

The Wilks p-value indicates that there is a significant relationship between group type and the dependent variables.

4.4.1 Results of ΔE and ΔL of each Group

The analysis that follows compares the results by group, independent of the sample type. The ΔE (change in colour) and ΔL (change in lightness) were compared by both control and experimental groups as indicated in Table 9.

Table 9. Comparison of ΔE and ΔL between control and experimental groups

Dependent Variable	Group	Mean	Std. Error	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
ΔE	Control	7.563[a]	0.096	7.370	7.755
	Experimental	7.444[a]	0.100	7.244	7.644
ΔL	Control	4.606[a]	0.129	4.349	4.863
	Experimental	5.121[a]	0.133	4.854	5.387

a. Based on modified population marginal mean.

To determine whether the one-way MANOVA was statistically significant, one needs to look at the "Sig". value for Wilks' lambda. It is noted from table 8 that the "Sig" value is 0.000, which means $p <$ 0.05. Therefore, it can be concluded that the multivariate effect of group is significant (Table 9).

The pairwise comparison indicates that there is a significant difference in the means between ΔL and the group types (Table 10), where p=0.007.

Table 10 : Mean difference between ΔL and the group types

Dependent Variable	(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.[d]	95% Confidence Interval for Difference[d] Lower Bound	95% Confidence Interval for Difference[d] Upper Bound
ΔE	Control	Experimental	0.119[a,b]	0.139	0.397	-0.159	0.396
	Experimental	Control	-0.119[a,b]	0.139	0.397	-0.396	0.159
ΔL	Control	Experimental	-0.514[a,b,*]	0.185	0.007	-0.885	-0.144
	Experimental	Control	0.514[a,b,*]	0.185	0.007	0.144	0.885

Based on estimated marginal means

*. The mean difference is significant at the 0.05 level.

a. An estimate of the modified population marginal mean (I).
b. An estimate of the modified population marginal mean (J).
d. Adjustment for multiple comparisons: Bonferroni.

The univariate analysis explored both the ΔE (change in colour) and ΔL (change in lightness). The univariate test indicates that there is a significant difference in the means between ΔL in contrast and error (Table 11), where p=0.007.

Table 11. Mean difference between ΔL in contrast and error

Dependent Variable		Sum of Squares	df	Mean Square	F	Sig.	Noncent. Parameter	Observed Power[a]
ΔE	Contrast	0.314	1	0.314	0.729	0.397	0.729	0.134
	Error	26.719	62	0.431				
ΔL	Contrast	5.912	1	5.912	7.702	0.007	7.702	0.780
	Error	47.595	62	0.768				

The F tests the effect of Group. This test is based on the linearly independent pairwise comparisons among the estimated marginal means.
a. Computed using alpha = 0.05

4.4.2 Effect of groups on ΔL and ΔE

These effects are independent of the other variables. The analysis that follows compares the results by group. The ΔE (change in colour) and ΔL (change in lightness) were compared for each sample group name. (Table 12).

Table 12. Comparison between ΔE and ΔL of each sample group

Dependent Variable	Sample Group Name	Mean	Std. Error	95% Confidence Interval	
				Lower Bound	Upper Bound
ΔE	CERCON	9.578[a]	0.109	9.359	9.796
	ZIRKON ZAHN	6.477[a]	0.123	6.231	6.723
	LAVA	7.019[a]	0.121	6.777	7.261
ΔL	CERCON	7.481[a]	0.146	7.189	7.772
	ZIRKON ZAHN	3.689[a]	0.164	3.360	4.017
	LAVA	4.220[a]	0.161	3.898	4.543

a. Based on modified population marginal mean.

The Wilks' Lambda test indicates a significant result where p=0.000. This is a statistical test used in multivariate analysis of variance (MANOVA) to test whether there are differences between the means of identified groups of subjects on a combination of dependant variables (Table 13).

Table 13. Differences between the means of identified groups

	Value	F	Hypothesis df	Error df	Sig.	Noncent. Parameter	Observed Power[c]
Pillai's trace	0.893	25.000	4.000	124.000	0.000	99.999	1.000
Wilks' lambda	0.117	58.674[a]	4.000	122.000	0.000	234.697	1.000
Hotelling's trace	7.464	111.963	4.000	120.000	0.000	447.854	1.000
Roy's largest root	7.453	231.041[b]	2.000	62.000	0.000	462.083	1.000

Each F tests the multivariate effect of Sample Group Name. These tests are based on the linearly independent pairwise comparisons among the estimated marginal means.

a. Exact statistic

b. The statistic is an upper bound on F that yields a lower bound on the significance level.

c. Computed using alpha = 0.05

The mean difference is significant between the group types for each of ΔL and ΔE ($p>0.05$), except for Zirkon Zahn (p=0.073) and Lava (p=0.073) for ΔL (Table 14).

Table 14. Difference between group types of ΔL and ΔE

Dependent Variable	(I) Sample Group Name	(J) Sample Group Name	Mean Difference (I-J)	Std. Error	Sig.[d]	95% Confidence Interval for Difference[d] Lower Bound	Upper Bound
ΔE	CERCON	ZIRKON ZAHN	3.101[*,b,c]	0.165	0.000	2.695	3.506
		LAVA	2.559[*,b,c]	0.163	0.000	2.157	2.960
	ZIRKON ZAHN	CERCON	-3.101[*,b,c]	0.165	0.000	-3.506	-2.695
		LAVA	-0.542[*,b,c]	0.173	0.008	-0.967	-0.118
	LAVA	CERCON	-2.559[*,b,c]	0.163	0.000	-2.960	-2.157
		ZIRKON ZAHN	0.542[*,b,c]	0.173	0.008	0.118	0.967
ΔL	CERCON	ZIRKON ZAHN	3.792[*,b,c]	0.220	0.000	3.251	4.333
		LAVA	3.260[*,b,c]	0.218	0.000	2.725	3.796
	ZIRKON ZAHN	CERCON	-3.792[*,b,c]	0.220	0.000	-4.333	-3.251
		LAVA	-0.532[b,c]	0.230	0.073	-1.099	0.035
	LAVA	CERCON	-3.260[*,b,c]	0.218	0.000	-3.796	-2.725
		ZIRKON ZAHN	0.532[b,c]	0.230	0.073	-0.035	1.099

Based on estimated marginal means

*. The mean difference is significant at the 0.05 level.

b. An estimate of the modified population marginal mean (I).

c. An estimate of the modified population marginal mean (J).

d. Adjustment for multiple comparisons: Bonferroni.

The results below (Table 15) investigate the effects of ΔL and ΔE on the contrast and error which indicates a significant effect on both variables. The univariate test indicates a significant result where *p*=0.000**.**

Table 15. Effects of ΔL and ΔE on the contrast and error

Dependent Variable		Sum of Squares	Df	Mean Square	F	Sig.	Noncent. Parameter	Observed Power[a]
ΔE	Contrast	181.392	2	90.696	210.454	0.000	420.908	1.000
	Error	26.719	62	.431				
ΔL	Contrast	279.362	2	139.681	181.957	0.000	363.914	1.000
	Error	47.595	62	.768				

The F tests the effect of Sample Group Name. This test is based on the linearly independent pairwise comparisons among the estimated marginal means.

a. Computed using alpha = 0.05

4.4.3 Firing

The results below (Table 16) investigate the effects of firing on ΔL and ΔE firing order which indicates a significant change in colour between the first and third firings and second and third firings respectively ($p<0.05$) (Appendix A).

Table 16. Effect of firing on ΔL and ΔE

				95% Confidence Interval	
Dependent Variable	Firing	Mean	Std. Error	Lower Bound	Upper Bound
ΔE	First	7.257[a]	0.120	7.017	7.496
	Second	7.196[a]	0.123	6.950	7.442
	Third	8.062[a]	0.120	7.823	8.302
ΔL	First	4.687[a]	0.160	4.367	5.006
	Second	4.552[a]	0.164	4.224	4.880
	Third	5.441[a]	0.160	5.121	5.761

a. Based on modified population marginal mean.

The pairwise comparison indicated that there is a significant difference in ΔE (change in colour) from the first and third firings and the second and third firing ($p<0.05$). All significant values are highlighted in Table 17. The pairwise comparison also indicated that there is a significant difference in ΔL (change in lightness) from the first and third firings and the second and third firing ($p<0.05$). This suggests that there is a noticeable change in colour and lightness from the first to third firing but not from the first and second firings.

Table 17. Differences in ΔE and ΔL after firings

Dependent Variable	(I) Firing	(J) Firing	Mean Difference (I-J)	Std. Error	Sig.[d]	95% Confidence Interval for Difference[d]	
						Lower Bound	Upper Bound
ΔE	First	Second	0061[a,b]	0.172	1.000	-0.362	0.484
		Third	-0.806[a,b,*]	0.169	0.000	-1.223	-0.388
	Second	First	-0.061[a,b]	0.172	1.000	-0.484	0.362
		Third	-0.867[a,b,*]	0.172	0.000	-1.289	-0.444
	Third	First	0.806[a,b,*]	0.169	0.000	0.388	1.223
		Second	0.867[a,b,*]	0.172	0.000	0.444	1.289
ΔL	First	Second	0.135[a,b]	0.229	1.000	-0.430	0.699
		Third	-0.754[a,b,*]	0.226	0.004	-1.311	-0.198
	Second	First	-0.135[a,b]	0.229	1.000	-0.699	0.430
		Third	-0.889[a,b,*]	0.229	0.001	-1.453	-0.325
	Third	First	0.754[a,b,*]	0.226	0.004	0.198	1.311
		Second	0.889[a,b,*]	0.229	0.001	0.325	1.453

Based on estimated marginal means

*. The mean difference is significant at the .05 level.

a. An estimate of the modified population marginal mean (I).

b. An estimate of the modified population marginal mean (J).

d. Adjustment for multiple comparisons: Bonferroni.

Table 18. Relationship between firing and dependant variables

The Wilks' *p*-value indicates that there is a significant relationship between firing and the dependant variables (colour and lightness).

	Value	F	Hypothesis df	Error df	Sig.	Noncent. Parameter	Observed Power[c]
Pillai's trace	0.346	6.480	4.000	124.000	0.000	25.920	0.989
Wilks' lambda	0.655	7.175[a]	4.000	122.000	0.000	28.699	0.995
Hotelling's trace	0.524	7.861	4.000	120.000	0.000	31.443	0.997
Roy's largest root	0.521	16.139[b]	2.000	62.000	0.000	32.279	0.999

Each F tests the multivariate effect of Firing. These tests are based on the linearly independent pairwise comparisons among the estimated marginal means.

a. Exact statistic

b. The statistic is an upper bound on F that yields a lower bound on the significance level.

c. Computed using alpha =0 .05

The results below (Table 19) compares the nature of contrast and error of ΔL and ΔE. The univariate test indicates a significant result where *p*=0.000 in both ΔL (change in lightness) and ΔE (change in colour) where (*p*<0.05).

Table 19. Comparison of contrast and error of ΔL and ΔE

Dependent Variable		Sum of Squares	df	Mean Square	F	Sig.	Noncent. Parameter	Observed Power[a]
ΔE	Contrast	13.885	2	6.943	16.110	0.000	32.219	0.999
	Error	26.719	62	0.431				
ΔL	Contrast	13.592	2	6.796	8.853	0.000	17.706	0.965
	Error	47.595	62	0.768				

The F tests the effect of Firing. This test is based on the linearly independent pairwise comparisons among the estimated marginal means.

a. Computed using alpha = 0.05

4.4.4 Shaded and unshaded

The results below (Table 20) compares the influence of shading on ΔL and ΔE.

Table 20. Comparison of influence of shading on ΔL and ΔE

Dependent Variable	Shaded / Unshaded	Mean	Std. Error	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
ΔE	Unshaded	12.990[a]	0.089	12.811	13.168
	Shaded	3.968[a]	0.099	3.769	4.167
ΔL	Unshaded	8.176[a]	0.119	7.938	8.414
	Shaded	2.771[a]	0.133	2.505	3.036

a. Based on modified population marginal mean.

The pairwise comparison indicated that there is a significant difference in ΔE (change in colour) and ΔL (change in lightness) for both shaded and unshaded samples, where *p*<0.05. All significant values are highlighted in Table 20. This suggests that there is a noticeable change in colour and lightness in both shaded and unshaded samples (Table 21).

Table 21. Difference in ΔE and ΔL for both shaded and unshaded samples

Dependent Variable	(I) Shaded / Unshaded	(J) Shaded / Unshaded	Mean Difference (I-J)	Std. Error	Sig.[d]	95% Confidence Interval for Difference[d]	
						Lower Bound	Upper Bound
ΔE	Unshaded	Shaded	9.022[*,b,c]	0.134	0.000	8.755	9.289
	Shaded	Unshaded	-9.022[*,b,c]	0.134	0.000	-9.289	-8.755
ΔL	Unshaded	Shaded	5.405[*,b,c]	0.178	0.000	5.048	5.762
	Shaded	Unshaded	-5.405[*,b,c]	0.178	0.000	-5.762	-5.048

Based on estimated marginal means

*. The mean difference is significant at the 0.05 level.

b. An estimate of the modified population marginal mean (I).

c. An estimate of the modified population marginal mean (J).

d. Adjustment for multiple comparisons: Bonferroni.

The Wilks' *p*-value indicates that there is a significant relationship between shaded and unshaded samples and the dependant variables (colour and lightness) (Table 22).

Table 22. Relationship between shaded and unshaded samples and the dependant variables

	Value	F	Hypothesis df	Error df	Sig.	Noncent. Parameter	Observed Power[b]
Pillai's trace	0.988	2544.566[a]	2.000	61.000	0.000	5089.132	1.000
Wilks' lambda	0.012	2544.566[a]	2.000	61.000	0.000	5089.132	1.000
Hotelling's trace	83.428	2544.566[a]	2.000	61.000	0.000	5089.132	1.000
Roy's largest root	83.428	2544.566[a]	2.000	61.000	0.000	5089.132	1.000

Each F tests the multivariate effect of Shaded / Unshaded. These tests are based on the linearly independent pairwise comparisons among the estimated marginal means.

a. Exact statistic

b. Computed using alpha = 0.05

4.4.5. Comparison of result to ΔL and ΔE

This section compares the result to ΔL and ΔE (Table 23). The result is A1, A2, B1, B2.

Table 23. Comparison of result to ΔL and ΔE

Dependent Variable	Result	Mean	Std. Error	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
ΔE	A1	3.250[a]	0.230	2.791	3.709
	A2	2.483[a]	0.219	2.046	2.921
	B1	12.990[a]	0.089	12.811	13.168
	B2	4.897[a]	0.124	4.649	5.144
ΔL	A1	1.964[a]	0.307	1.351	2.577
	A2	1.389[a]	0.292	0.805	1.973
	B1	8.176[a]	0.119	7.938	8.414
	B2	3.700[a]	0.165	3.370	4.030

a. Based on modified population marginal mean.

The pairwise comparison (Table 24) indicated that there is a significant difference in ΔE (change in colour) and ΔL (change in lightness) and the result. The following were found significant:

A1 with B1 and B2 ($p<0.05$)

A2 with B1 and B2 ($p<0.05$)

B1 with A1, A2 and B2 ($p<0.05$)

B2 with A1, A2 and B1 ($p<0.05$)

Table 24. Difference in ΔE, ΔL and the result

Dependent Variable	(I) Result	(J) Result	Mean Difference (I-J)	Std. Error	Sig.[d]	95% Confidence Interval for Difference[d] Lower Bound	95% Confidence Interval for Difference[d] Upper Bound
ΔE	A1	A2	0.767[a,b]	0.317	0.112	-0.098	1.631
		B1	-9.740[a,b,*]	0.246	0.000	-10.411	-9.068
		B2	-1.647[a,b,*]	0.261	0.000	-2.358	-0.935
	A2	A1	-0.767[a,b]	0.317	0.112	-1.631	0.098
		B1	-10.506[a,b,*]	0.236	0.000	-11.150	-9.862
		B2	-2.413[a,b,*]	0.251	0.000	-3.099	-1.728
	B1	A1	9.740[a,b,*]	0.246	0.000	9.068	10.411
		A2	10.506[a,b,*]	0.236	0.000	9.862	11.150
		B2	8.093[a,b,*]	0.153	0.000	7.677	8.509
	B2	A1	1.647[a,b,*]	0.261	0.000	0.935	2.358
		A2	2.413[a,b,*]	0.251	0.000	1.728	3.099
		B1	-8.093[a,b,*]	0.153	0.000	-8.509	-7.677

ΔL	A1	A2	0.575[a,b]	0.423	1.000	-0.579	1.729
		B1	-6.212[a,b,*]	0.329	0.000	-7.108	-5.315
		B2	-1.736[a,b,*]	0.348	0.000	-2.685	-0.786
	A2	A1	-0.575[a,b]	0.423	1.000	-1.729	0.579
		B1	-6.787[a,b,*]	0.315	0.000	-7.647	-5.927
		B2	-2.311[a,b,*]	0.336	0.000	-3.226	-1.397
	B1	A1	6.212[a,b,*]	0.329	0.000	5.315	7.108
		A2	6.787[a,b,*]	0.315	0.000	5.927	7.647
		B2	4.476[a,b,*]	0.204	0.000	3.921	5.031
	B2	A1	1.736[a,b,*]	0.348	0.000	0.786	2.685
		A2	2.311[a,b,*]	0.336	.000	1.397	3.226
		B1	-4.476[a,b,*]	0.204	.000	-5.031	-3.921

Based on estimated marginal means

*. The mean difference is significant at the 0.05 level.

a. An estimate of the modified population marginal mean (I).

b. An estimate of the modified population marginal mean (J).

d. Adjustment for multiple comparisons: Bonferroni.

The Wilks' *p*-value (Table 25) indicates that there is a significant relationship between the result and the dependant variables (colour and lightness).

Table 25. Relationship between the result and the dependant variables

	Value	F	Hypothesis df	Error df	Sig.	Noncent. Parameter	Observed Power[c]
Pillai's trace	1.198	30.903	6.000	124.000	0.000	185.416	1.000
Wilks' lambda	0.009	191.004[a]	6.000	122.000	0.000	1146.022	1.000
Hotelling's trace	84.585	845.850	6.000	120.000	0.000	5075.103	1.000
Roy's largest root	84.319	1742.590[b]	3.000	62.000	0.000	5227.771	1.000

Each F tests the multivariate effect of Result. These tests are based on the linearly independent pairwise comparisons among the estimated marginal means.

a. Exact statistic

b. The statistic is an upper bound on F that yields a lower bound on the significance level.

c. Computed using alpha = 0.05

4.5 Relationships to the dependent variables

The GLM Multivariate procedure is based on the general linear model. The factors and covariates presented here are assumed to have linear relationships to the dependent variables.

The intercept, Group, Sample Group Name and Result all had a significant Wilks' Lambda reading (*p*=0.000). Firing was the only effect that had no significant Wilks' Lambda, where *p*=0.112.

All significant Wilks' Lambda imply that a combination of the variables did have a significant interaction between the variables (Table 26). Significant Wilks' Lambda was found for the following:

- Group * Result (*p*=0.001)
- Sample Group Name * Firing (*p*=0.000)
- Firing * Result (*p*=0.000)
- Group * Sample Group Name * Firing (*p*=0.000)
- Group * Firing * Result (*p*=0.000)

There was no significant interaction between the following variables (insignificant Wilks' Lambda):

- Group * Sample Group Name (*p*=0.098)
- Group * Firing (*p*=0.053
- Sample Group Name * Result (*p*=0.443)

Table 26. Interaction between variables

	Roy's Largest Root	174.238	5314.271[b]	2.000	61.000	0.000	10628.541	1.000		
Group	Pillai's Trace	0.326	Effect	Value	F	Hypothesis df	Error df	Sig.	Noncent. Parameter	Observed Power[d]
	Wilks' Lambda	0.674	Intercept	Pillai's Trace	0.994	5314.271[b]	2.000	61.000	0.000	10628.541	1.000
	Hotelling's Trace	0.484	14.751[b]	Wilks' Lambda	0.006	5314.271[b]	2.000	61.000	0.000	10628.541	1.000
	Roy's Largest Root	0.484	14.751[b]	Hotelling's Trace	174.238	5314.271[b]	2.000	61.000	0.000	10628.541	1.000
Sample_Group_Name	Pillai's Trace	0.951	28.086	4.000	124.000	0.000	112.345	1.000		
	Wilks' Lambda	0.194	38.739[b]	4.000	122.000	0.000	154.957	1.000		
	Hotelling's Trace	3.408	51.115	4.000	120.000	0.000	204.459	1.000		
	Roy's Largest Root	3.173	98.349[c]	2.000	62.000	0.000	196.698	1.000		
Firing	Pillai's Trace	0.117	1.919	4.000	124.000	0.111	7.675	0.566		
	Wilks' Lambda	0.885	1.915[b]	4.000	122.000	0.112	7.660	0.565		
	Hotelling's Trace	0.127	1.911	4.000	120.000	0.113	7.643	0.563		
	Roy's Largest Root	0.107	3.330[c]	2.000	62.000	0.042	6.661	0.610		
Result	Pillai's Trace	0.291	5.280	4.000	124.000	0.001	21.118	0.967		
	Wilks' Lambda	0.713	5.629[b]	4.000	122.000	0.000	22.514	0.976		
	Hotelling's Trace	0.398	5.968	4.000	120.000	0.000	23.874	0.982		
	Roy's Largest Root	0.384	11.912[c]	2.000	62.000	0.000	23.823	0.993		
Group * Sample_Group_Name	Pillai's Trace	0.120	1.981	4.000	124.000	0.101	7.926	0.581		
	Wilks' Lambda	0.880	2.006[b]	4.000	122.000	0.098	8.025	0.587		
	Hotelling's Trace	0.135	2.030	4.000	120.000	0.095	8.118	0.593		
	Roy's Largest Root	0.131	4.054[c]	2.000	62.000	0.022	8.109	0.701		
Group * Firing	Pillai's Trace	0.145	2.417	4.000	124.000	0.052	9.669	0.680		
	Wilks' Lambda	0.859	2.411[b]	4.000	122.000	0.053	9.643	0.679		
	Hotelling's Trace	0.160	2.403	4.000	120.000	0.053	9.613	0.677		

	Roy's Largest Root	0.128	3.973[c]	2.000	62.000	0.024	7.946	0.692
Group * Result	Pillai's Trace	0.214	8.294[b]	2.000	61.000	0.001	16.587	0.954
	Wilks' Lambda	0.786	8.294[b]	2.000	61.000	0.001	16.587	0.954
	Hotelling's Trace	0.272	8.294[b]	2.000	61.000	0.001	16.587	0.954
	Roy's Largest Root	0.272	8.294[b]	2.000	61.000	0.001	16.587	0.954
Sample_Group_Name * Firing	Pillai's Trace	0.560	6.022	8.000	124.000	0.000	48.177	1.000
	Wilks' Lambda	0.448	7.524[b]	8.000	122.000	0.000	60.195	1.000
	Hotelling's Trace	1.212	9.093	8.000	120.000	0.000	72.745	1.000
	Roy's Largest Root	1.198	18.562[c]	4.000	62.000	0.000	74.246	1.000
Sample_Group_Name * Result	Pillai's Trace	0.026	0.825[b]	2.000	61.000	0.443	1.651	0.185
	Wilks' Lambda	0.974	0.825[b]	2.000	61.000	0.443	1.651	0.185
	Hotelling's Trace	0.027	0.825[b]	2.000	61.000	0.443	1.651	0.185
	Roy's Largest Root	0.027	0.825[b]	2.000	61.000	0.443	1.651	0.185
Firing * Result	Pillai's Trace	0.484	4.954	8.000	124.000	0.000	39.631	0.998
	Wilks' Lambda	0.526	5.781[b]	8.000	122.000	0.000	46.247	1.000
	Hotelling's Trace	0.882	6.618	8.000	120.000	0.000	52.947	1.000
	Roy's Largest Root	0.860	13.328[c]	4.000	62.000	0.000	53.314	1.000
Group * Sample_Group Name * Firing	Pillai's Trace	0.701	8.373	8.000	124.000	0.000	66.984	1.000
	Wilks' Lambda	0.403	8.778[b]	8.000	122.000	0.000	70.226	1.000
	Hotelling's Trace	1.224	9.178	8.000	120.000	0.000	73.425	1.000
	Roy's Largest Root	0.952	14.753[c]	4.000	62.000	0.000	59.011	1.000
Group * Firing * Result	Pillai's Trace	0.341	6.361	4.000	124.000	0.000	25.446	0.988
	Wilks' Lambda	0.660	7.041[b]	4.000	122.000	0.000	28.162	0.994
	Hotelling's Trace	0.514	7.710	4.000	120.000	0.000	30.842	0.997
	Roy's Largest Root	0.512	15.878[c]	2.000	62.000	0.000	31.757	0.999

4.5.1 Results of between-subjects effects of dependant and independent variables

To determine how the dependent variables differ from the independent variable, the tests of between-subjects effects was investigated (Table 27) and the following was observed:

- The combination of Group and Result has a statistically significant effect on both ΔE (F =16.091) $p < 0.0005$; and ΔL (F =11.986) $p < 0.0005$. There is a level of acceptance of the result as the observed power is very large (and close to 1).
- The combination of Sample Group Name and Firing has a statistically significant effect on both ΔE (F =17.339) $p < 0.0005$; and ΔL (F =14.010) $p < 0.0005$. There is a level of acceptance of the result as the observed power is 1.
- The combination of Group, Sample Group Name and Firing has a statistically significant effect on both ΔE (F =11.076) $p < 0.0005$; and ΔL (F =14.515) $p < 0.0005$. There is a level of acceptance of the result as the observed power is 1.
- The combination of Group, Firing and Result has a statistically significant effect on both ΔE (F =15.517) $p < 0.0005$; and ΔL (F =10.372) $p < 0.0005$. There is a level of acceptance of the result as the observed power is very large (ΔE: 0.999 and ΔL: 0.984)

Table 27. Tests of between-subjects effects of dependant and independent variables

Source	Dependent Variable	Type III Sum of Squares	Df	Mean Square	F	Sig.	Noncent. Parameter	Observed Power[c]
Corrected Model	ΔE	2356.083[a]	45	52.357	121.492	0.000	5467.151	1.000
	ΔL	1155.591[b]	45	25.680	33.452	0.000	1505.340	1.000
Intercept	ΔE	4254.972	1	4254.972	9873.408	0.000	9873.408	1.000
	ΔL	1838.970	1	1838.970	2395.548	0.000	2395.548	1.000
Group	ΔE	8.916	1	8.916	20.690	0.000	20.690	0.994
	ΔL	22.179	1	22.179	28.891	0.000	28.891	1.000
Sample_Group_Name	ΔE	40.264	2	20.132	46.715	0.000	93.430	1.000
	ΔL	150.317	2	75.158	97.906	0.000	195.811	1.000
Firing	ΔE	2.836	2	1.418	3.291	0.044	6.582	0.604
	ΔL	3.541	2	1.771	2.307	0.108	4.613	0.451
Result	ΔE	8.987	2	4.493	10.427	0.000	20.853	0.985
	ΔL	3.585	2	1.793	2.335	0.105	4.671	0.456
	ΔE	0.141	2	0.070	0.164	0.850	0.327	0.074

Group * Sample_Group_Name	ΔL	3.669	2	1.835	2.390	0.100	4.779	0.465
Group * Firing	ΔE	2.232	2	1.116	2.590	0.083	5.179	0.498
	ΔL	1.533	2	0.766	0.998	0.374	1.997	0.216
Group * Result	ΔE	6.934	1	6.934	16.091	0.000	16.091	0.977
	ΔL	9.201	1	9.201	11.986	0.001	11.986	0.926
Sample_Group_Name * Firing	ΔE	29.890	4	7.472	17.339	0.000	69.358	1.000
	ΔL	43.021	4	10.755	14.010	0.000	56.041	1.000
Sample_Group_Name * Result	ΔE	0.258	1	0.258	0.598	0.442	0.598	0.119
	ΔL	1.260	1	1.260	1.641	0.205	1.641	0.243
Firing * Result	ΔE	21.058	4	5.265	12.216	0.000	48.865	1.000
	ΔL	9.931	4	2.483	3.234	0.018	12.936	0.802
Group * Sample_Group_Name * Firing	ΔE	19.093	4	4.773	11.076	0.000	44.304	1.000
	ΔL	44.570	4	11.142	14.515	0.000	58.059	1.000
Group * Firing * Result	ΔE	13.374	2	6.687	15.517	0.000	31.033	0.999
	ΔL	15.924	2	7.962	10.372	0.000	20.743	0.984

a. R Squared =0.989 (Adjusted R Squared = 0.981)

b. R Squared = 0.960 (Adjusted R Squared = 0.932)

c. Computed using alpha = 0.05

4.6 Estimated marginal means for ΔE

4.6.1 Estimated marginal means for ΔE (Group)

The plots below (Figure 13) indicate the estimated marginal means for ΔE

It is noted that the experimental group has a lower mean value than the control group. A possible reason for this finding would be that the control group was not exposed to firing. The experimental group was exposed to firing, and from Figure 14 it can be noted that there were several different colour changes during the firing process.

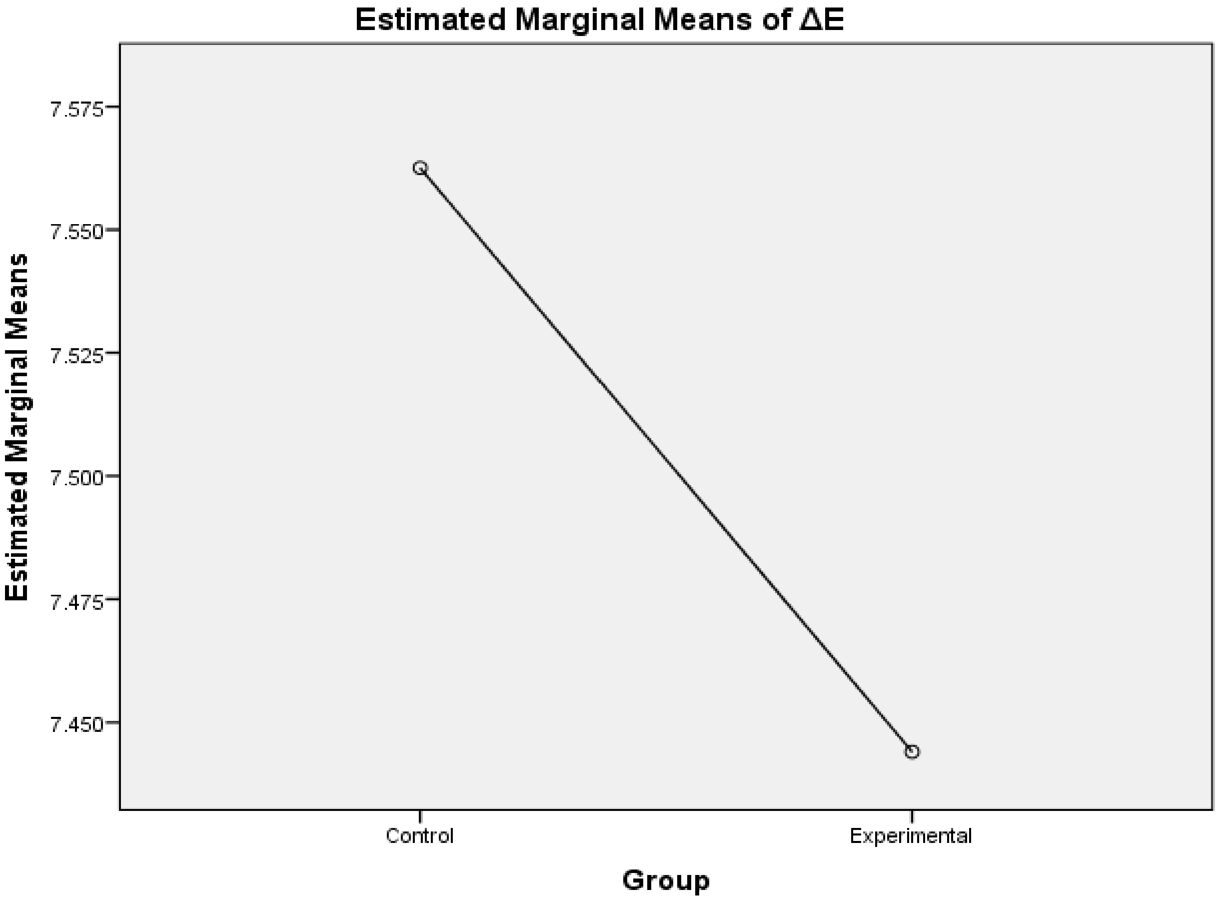


Figure 13. Estimated Marginal Means of ΔE

4.6.2 Estimated marginal means for ΔE (Sample Group Name)

Cercon had the highest mean and Zirkon Zahn had the lowest mean for ΔE (Figure 14). This suggests that Cercon is the most stable as there was the least amount of colour change that occurred when compared to Zirkon Zahn and Lava. Zirkon Zahn would be the least stable as there was the most colour change at the end of the experiment. This suggests that Zirkon Zahn is the least stable.

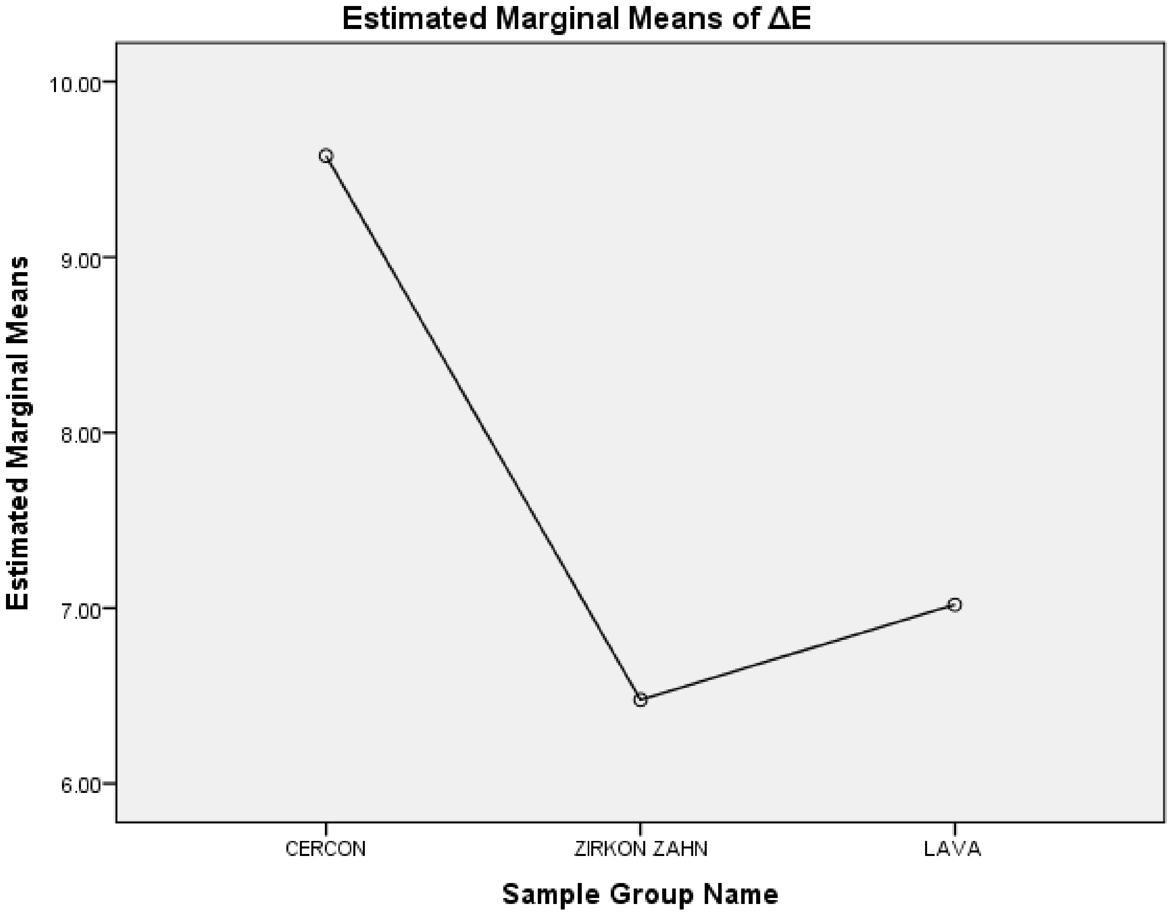


Figure 14. Estimated Marginal Means ΔE (Sample Group Name)

4.6.3 Estimated marginal means for ΔE (Firing)

The highest mean was seen after the third firing. This suggest that there is a noticeable ΔE from the first to the third firing as well as from the second to the third firing. There was no significant change from the first and second firings (Figure 15).

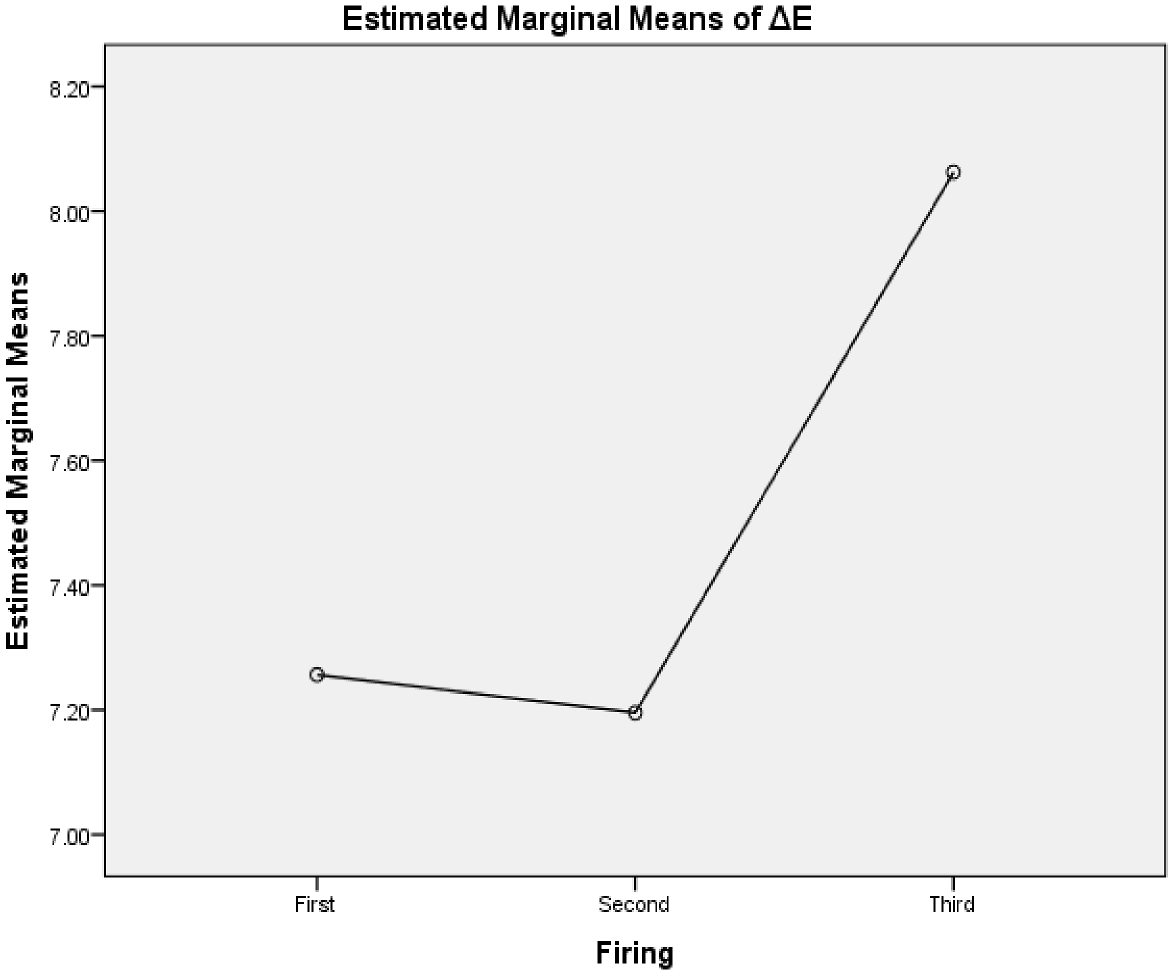


Figure 15. Estimated Marginal Means of ΔE (Firing)

4.6.4 Estimated marginal means for ΔE (Shaded / Unshaded)

The unshaded samples had a higher mean than the shaded samples. This suggests that the unshaded samples were more stable than the shaded samples as there was no colour change noted (Figure 16).

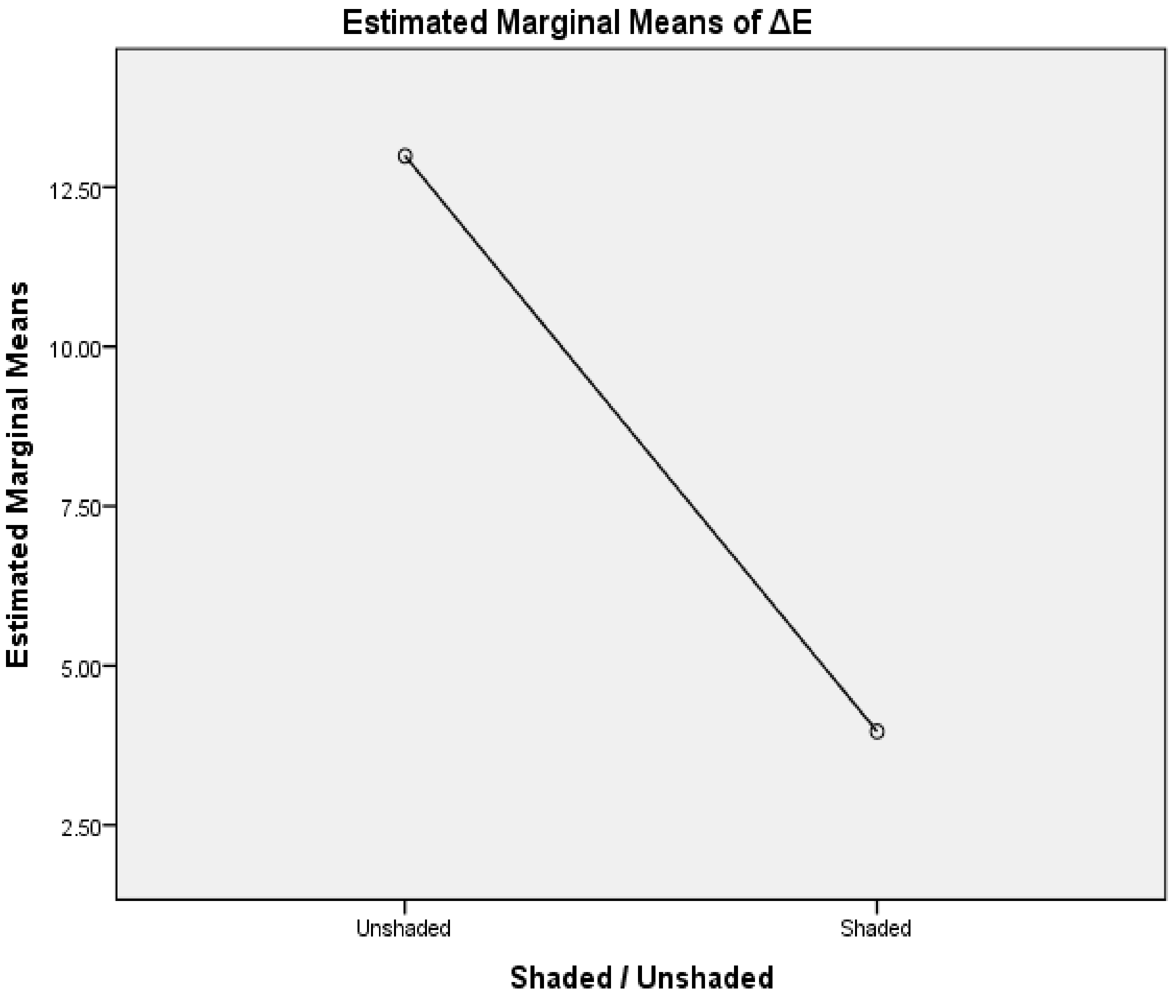


Figure 16. Estimated Marginal means for ΔE (Shaded/Unshaded)

4.6.5 Estimated marginal means for ΔE (Result)

All unshaded specimens were B1. B1 had the highest mean. There was no change in colour which suggests that the unshaded specimens were the most stable (Figure 17).

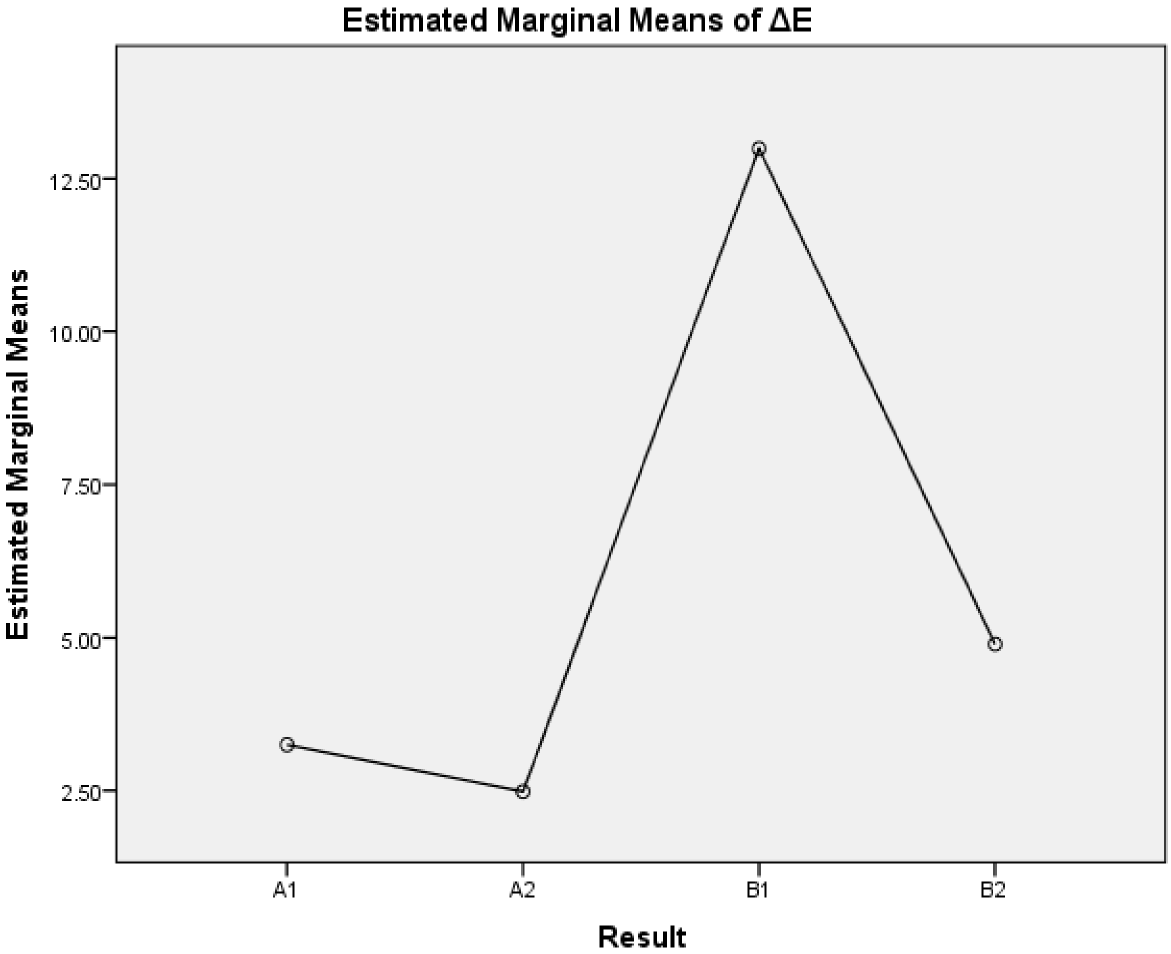


Figure 17. Estimated Marginal Means of ΔE (Result)

4.7 Estimated marginal means for ΔL

4.7.1 Estimated marginal means for ΔL (Group)

The estimated marginal mean value for ΔL is greater in the experimental group than the control group which indicates that the experimental group is lighter than the control group (Figure 18). The greater the L* value is, the lighter the specimen.

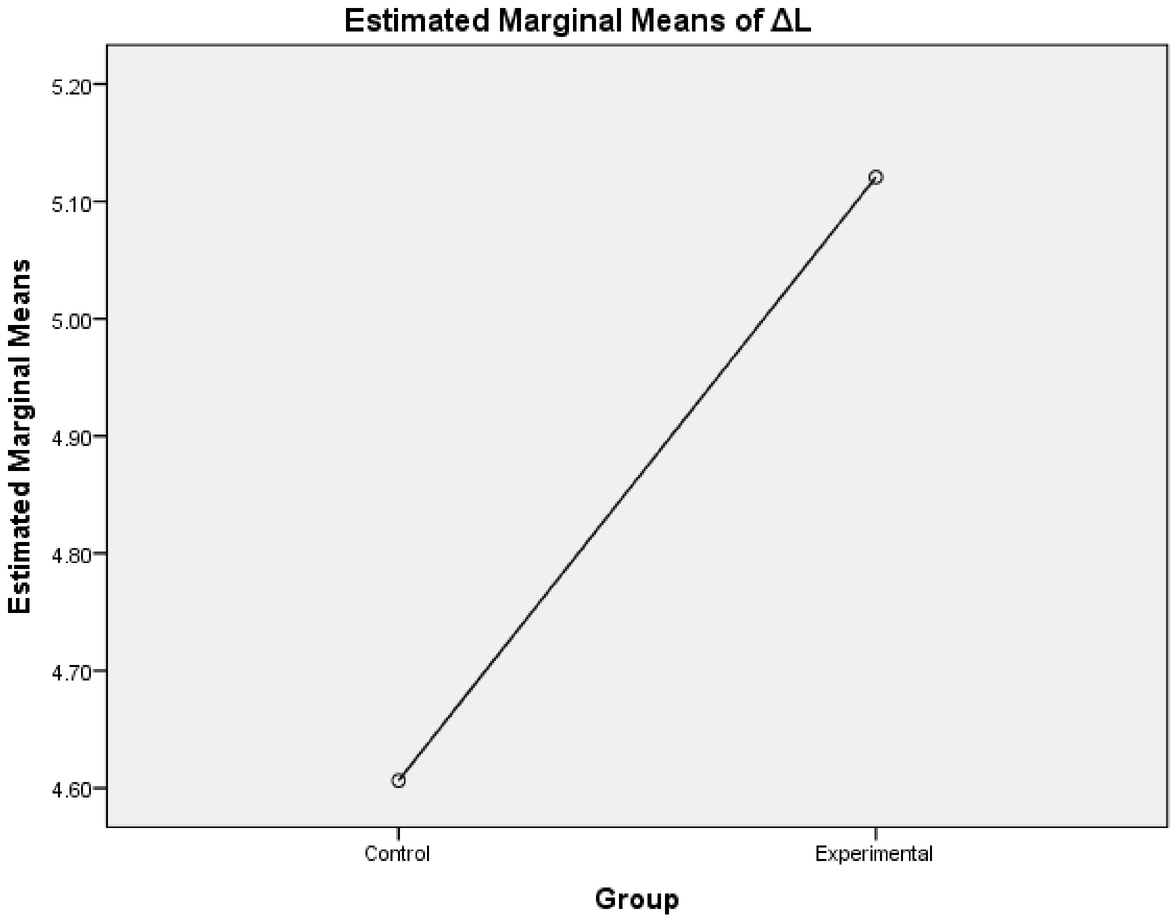


Figure 18. Estimated Marginal Means of ΔL (Group)

4.7.2 Estimated marginal means for ΔL (Sample Group Name)

Cercon had the highest mean and Zirkon Zahn had the lowest mean for ΔL (Figure 19). This suggests that Cercon is the most stable as there was the least amount of change in lightness that occurred when compared to Zirkon Zahn and Lava. Zirkon Zahn would be the least stable as there was the most change in lightness at the end of the experiment. This suggests that Zirkon Zahn is the least stable.

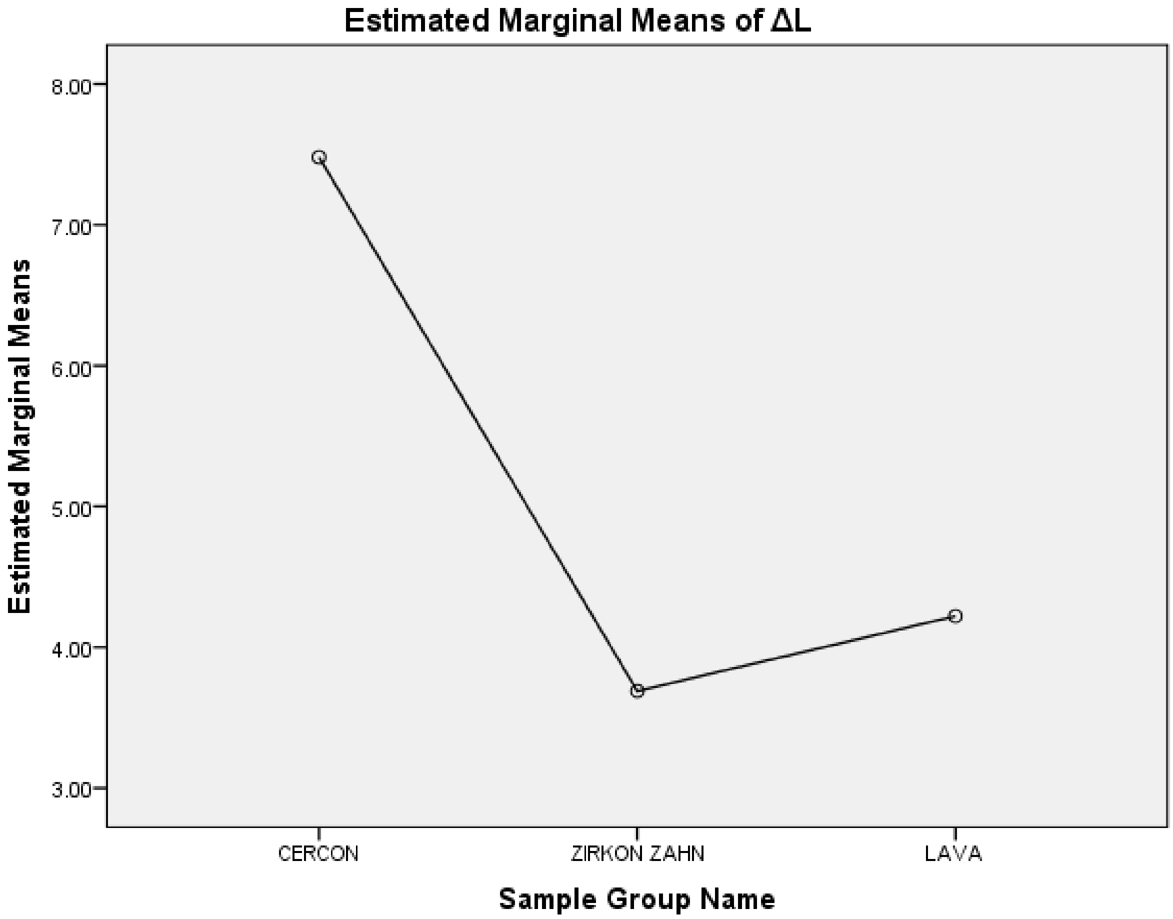


Figure 19. Estimated Marginal Means of ΔL (Sample Group Name)

4.7.3 Estimated marginal means for ΔL (Firing)

The highest mean was seen after the third firing. This suggest that there is a noticeable ΔL from the first to the third firing as well as from the second to the third firing. There was no significant change from the first to the second firings (Figure 20).

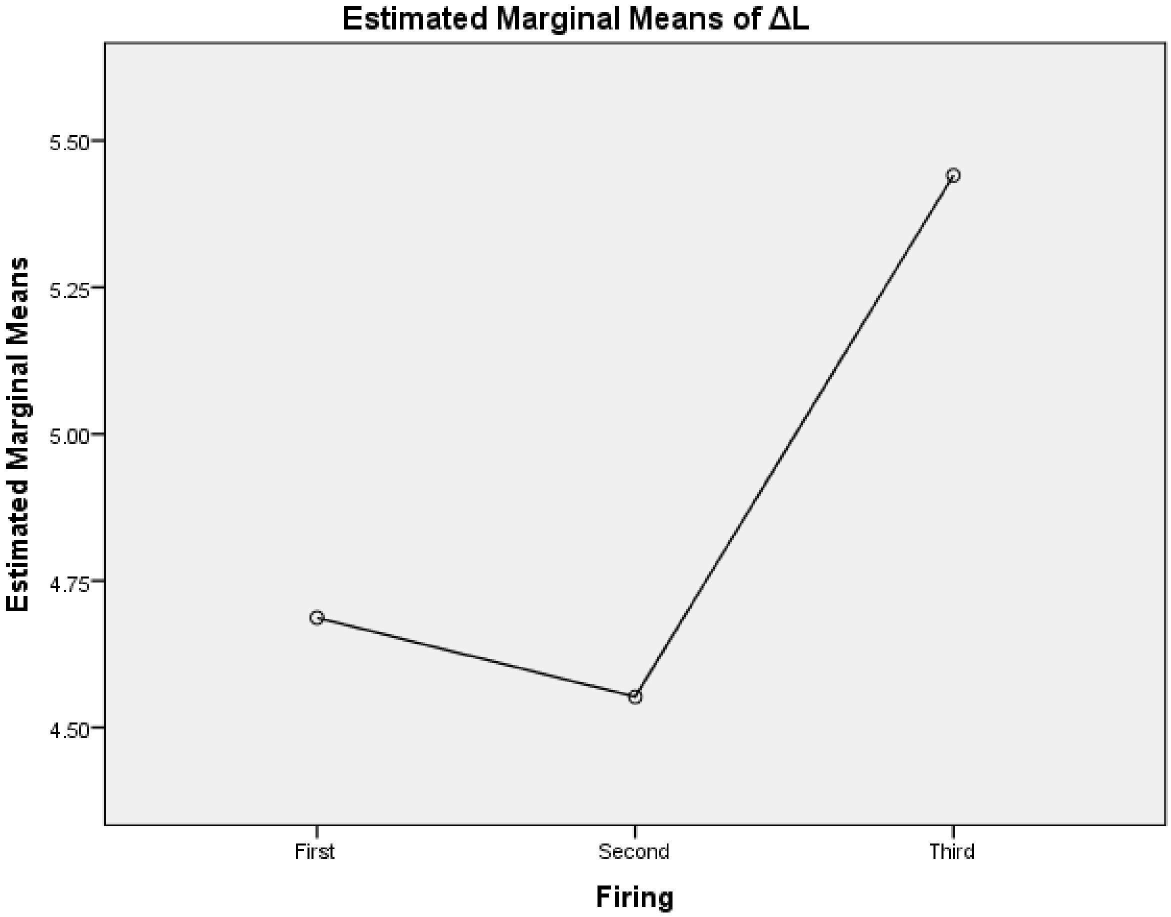


Figure 20. Estimated Marginal Means of ΔL (Firing)

4.7.4 Estimated marginal means for ΔL (Shaded / Unshaded)

The unshaded samples had a higher mean than the shaded samples (Figure 21). This suggests that the unshaded samples were more stable than the shaded samples as there was no colour change noted. This can also be seen in Figure 21.

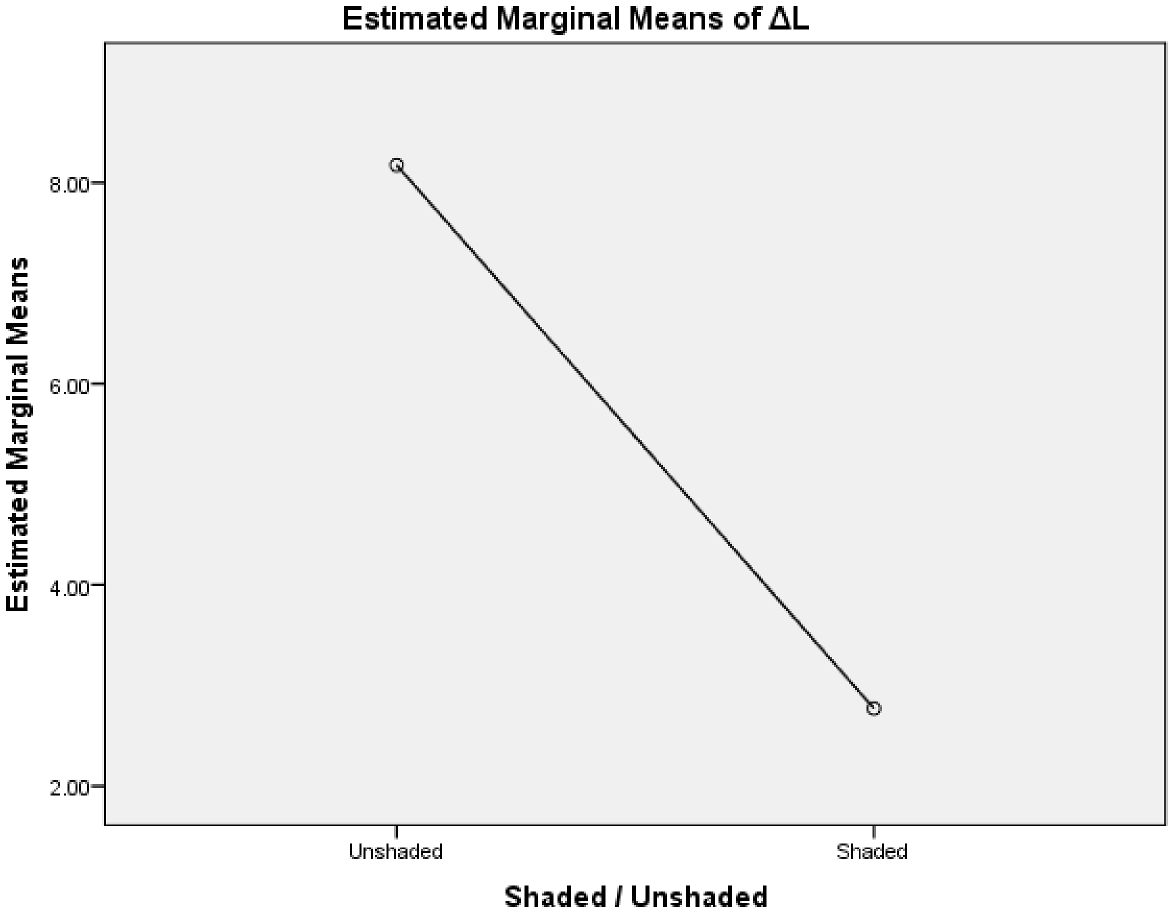


Figure 21. Estimated Marginal Means of ΔL (Shaded/Unshaded)

4.7.5 Estimated marginal means for ΔL (Result)

All unshaded specimens were B1. B1 had the highest mean. There was no change in lightness which suggests that the unshaded specimens were the most stable (Figure 22).

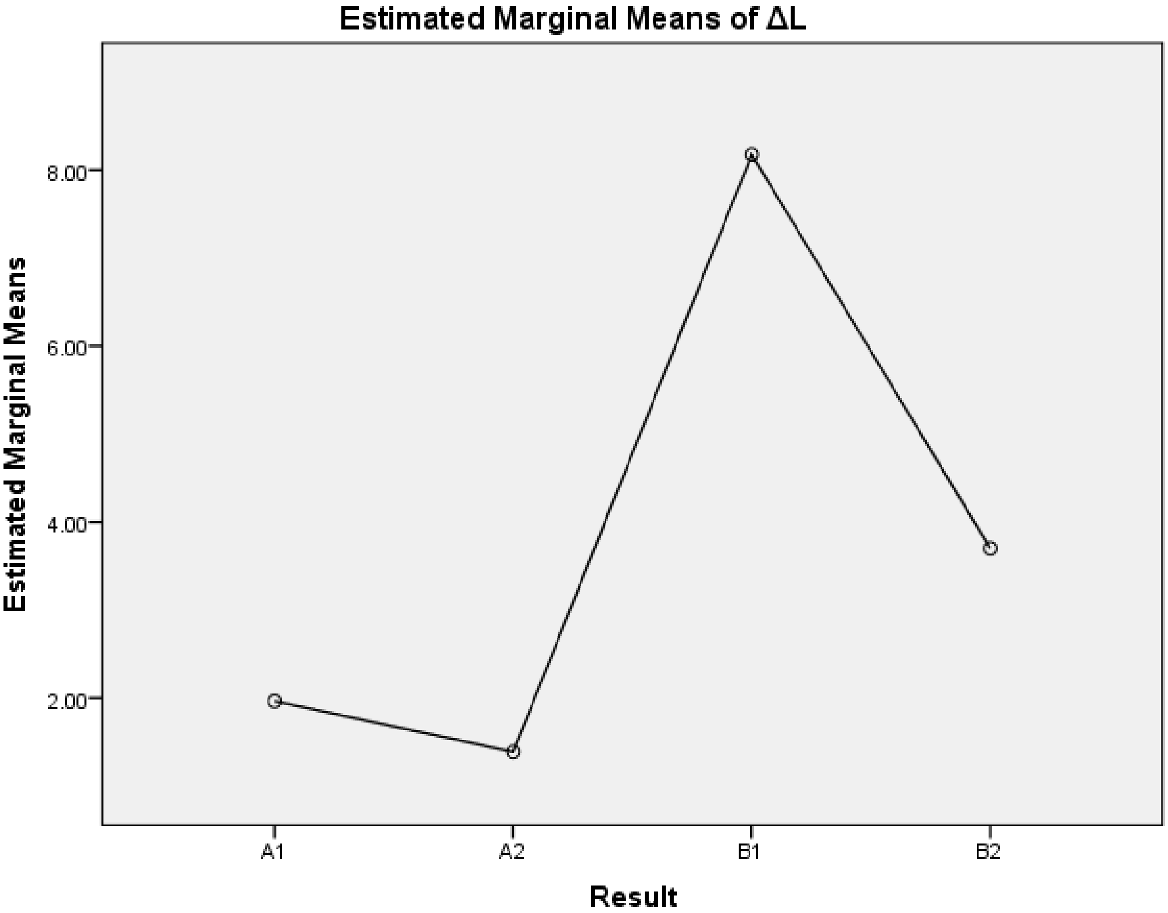


Figure 22. Estimated Marginal Means of ΔL (Result)

4.8 SEM Analysis

The three groups had different elemental components and grain appearances. Lava™ and Cercon® had similar grain sizes whereas Zirkon Zahn® had a slightly larger grain size. Although grain growth was visible, the present study found no major change in the grain size of all three dental zirconia systems after numerous firing cycles up to 800°C. This was done with the actual measurement of individual grains provided during SEM analysis.

Cercon®

The microstructure contained fine grains with some larger grains. Grain growth was visible at a magnitude of 10000 KX and after the third firing (Figure 23-24). After measurement, the mean grain size was 0.11±0.04 µm, while large grains were approximately 0.2-0.3 µm. The grains in Cercon specimens seemed to have fused grains and a coalesced pattern.

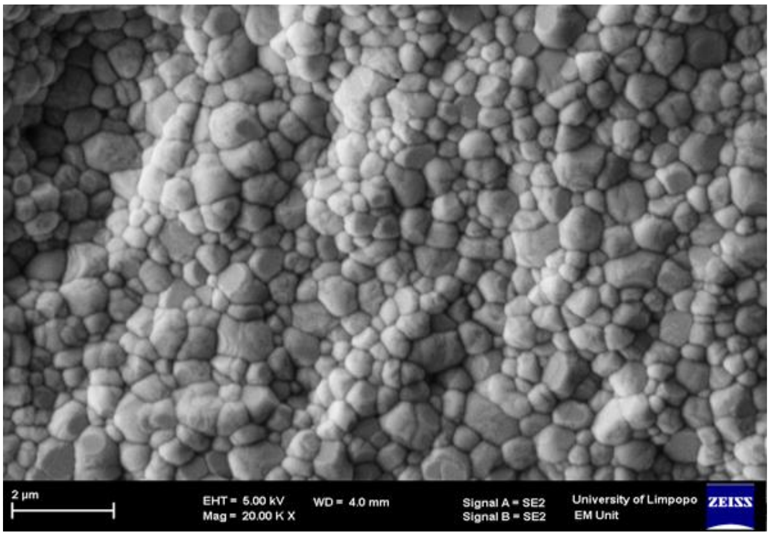


Figure 23. Scan electron micrograph of Cercon before firing

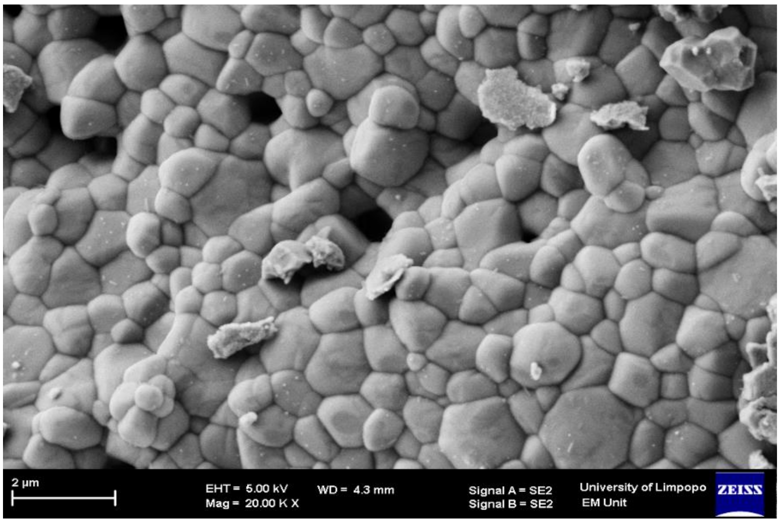


Figure 24. Scan electron micrograph of Cercon after third firing

Zirkon Zahn®

Zirkon Zahn had a slightly larger grain size than Cercon and Lava. Microstructure of the Zirkon Zahn specimen contained large grains with some fine grains as well (Figure 25-26). Dark spots in certain areas may be inconsistent infiltration of shading liquid. The measurement of the mean grain size of fired and unfired Zirkon Zahn specimens was 0.11±0.13 µm, while some large grains were approximately 0.2 µm.

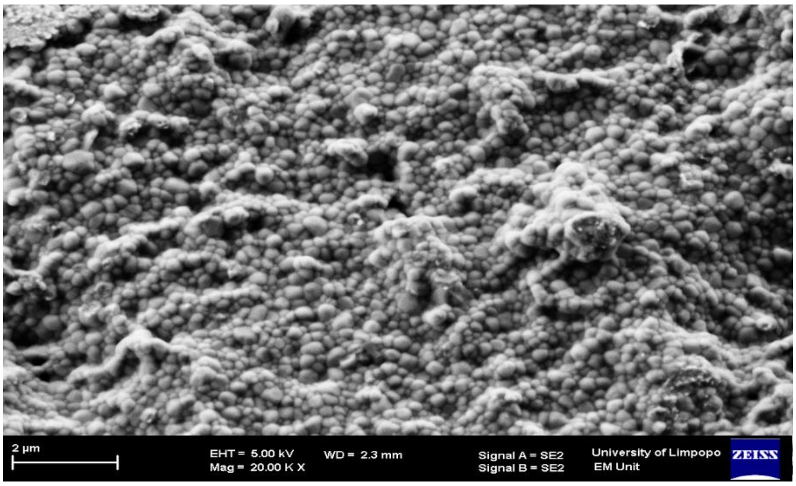


Figure 25. Scan electron micrograph of Zirkon Zahn before firing

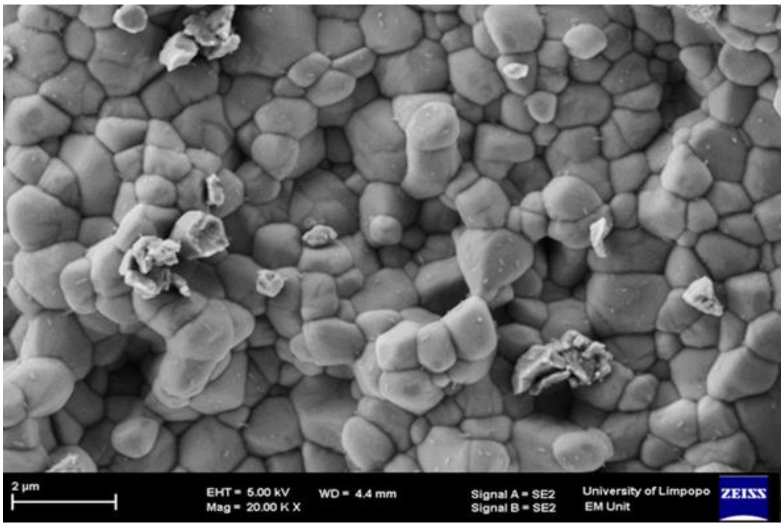


Figure 26. Scan electron micrograph of Zirkon Zahn after third firing

***Lava*™**

Figure 27 shows the fine microstructures of unfired Lava specimens. The microstructure of Lava copings contained predominantly fine grains with some larger grains. The grain appearance was rounded and separated. The mean grain size of the unfired Lava specimen was 0.10±0.042 µm. The larger grains were about 0.2 µm. The Lava specimen after the third firing (Figure 28), had the mean grain size of 0.11±0.05 µm while the large grains were approximately 0.2-0.3 µm.

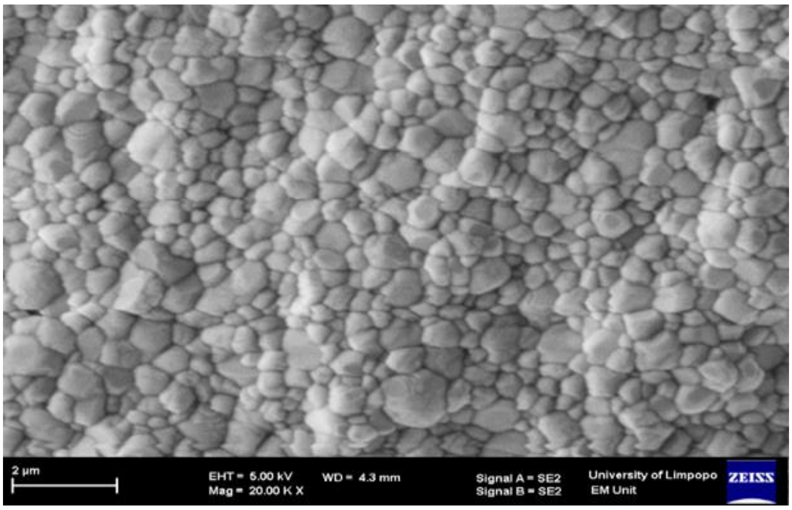


Figure 27. Scan electron micrograph of Lava before firing

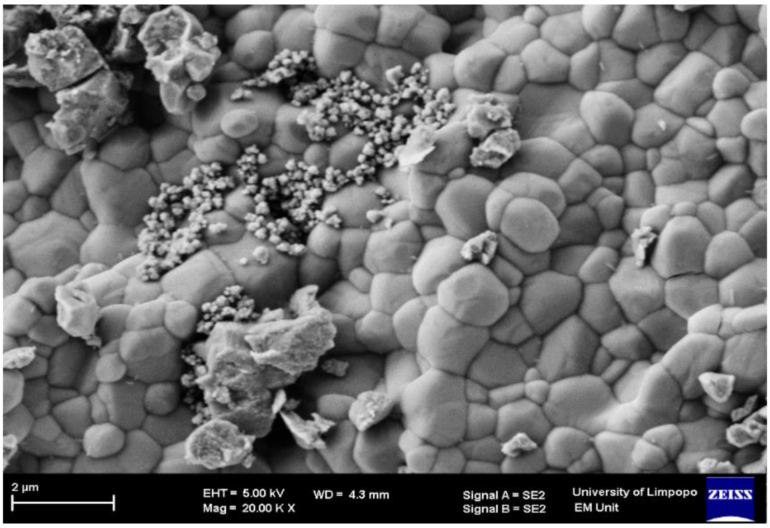


Figure 28. Scan electron micrograph of Lava after third firing

CHAPTER FIVE

DISCUSSION

5.1 Introduction

Multiple studies have been conducted on the effect of numerous firings on different zirconia materials, (Celik *et al.*, 2008; Baldissara *et al.*, 2010). However, none have been conducted on the Lava, Cercon and Zirkon Zahn systems respectively. There is a paucity of studies determining the colour stability of zirconia systems. The studies previously done, such as the study by Celik *et al.* (2008) did not focus on core structures itself but disk shaped core material with ceramic application. This study included cores with a uniform thickness of 0.5mm. A consistent thickness allows for a defined condition under which the colour differences can be quantified and brands can be compared (Denry *et al.*, 2008). The cores were requested from each manufacturer which were Lava, Cercon and Zirkon Zahn. The cores were divided into shaded (A2) and unshaded (white). The shading method was identified as a variable that may affect the intensity of the pigmentation since it had been highlighted that the concentration of the colouring solution affects the final shade (Denry *et al.*, 2008). In the present study, the specimens received from the manufacturer were all tested for their initial colour using a spectrophotometer, before being exposed to firing cycles. Vita states that when using a fibrous pad, the temperature should be raised by 20^{0}C higher, since 20^{0}C is absorbed by the fibrous pad and may result in the firing temperature being much lower than expected (VITA Zahnfabrik, Bad Säckingen, Germany). This could lead to zirconia copings being underbaked.

It is important to note that all specimens were received shaded and sintered by the respective manufacturers. There are different ways of colouring zirconia frameworks as mentioned in chapter 2. The method and duration during which samples were left in shading liquid was unknown on receipt. The results of the shaded cores varied between the shades A1, A2 and B2 which proves that concentration of shading solution does play a role in colour differences. The duration during which the specimens were left in the shading liquid and perhaps different manufacturers who use different methods may have been a contributing factor to colour variation.

The spectrophotometer was the most suitable to this study as it provides either an overall shade which was used in this study or the shade can be broken down into three distinct areas, cervical, middle and incisal. The system offers detailed shade information in each of these areas if required and will provide a mathematical analysis resulting in a ΔE value (Chu *et al*., 2010). Virtual shade verification can be performed with this system so that shades can be guaranteed prior to time-consuming patient visits. It is important that the shade analysis is performed on the correct (upper / lower) orientation, because the system makes a direct comparison of corresponding area on the tooth and on the shade guide. As the shade guides have different colour in cervical / middle / incisal regions, it's mandatory to use the correct tooth orientation (Sahin *et al.*, 2010). A mouth simulator was used during the spectrophotometer reading which allows one to view the restoration under the same lighting conditions that are present in the oral cavity. Due to the high costs involved with the SEM analysis, a limited number of copings were analysed.

5.2 Sample size

A total number of 54 zirconia copings were used. Eighteen specimens for each of the zirconia groups were manufactured. Nine specimens in each sample group were shaded using the A2 shade and nine were unshaded. Zirconia cores are ideally shaded in order to mimic the dentin. The study conducted by Celik *et al*. (2008), and on which the present study is based consisted of 20 specimens.

5.3 Blinding of the study

The coding of each specimen served the following purpose:

- The specimens were easily identified during the various testing stages
- The data captured by the spectrophotometer (SpectroShade Micro (MHT, Niederhasli, Switzerland) was encoded due to this research being a blind study.

5.4 Results of the sample composition

All unshaded specimens had a result type of B1 as shown in Figure 12 and Table 6. It is important to note that unshaded specimens which were B1 in all sample groups remained unchanged. However, B1 zirconia does not lead to an optimal aesthetic result and shading the zirconia is necessary according to Komine *et al*. (2012); Happe *et al*. (2013); Ishikawa-Nagai *et al.* (2013), Kurtulmus-Yilmaz and Ulusoy (2014) and Kaya (2013).

The Cercon results for both the control and experimental groups reveal the same result patterns as seen in the B2 shade maintained before and after firing as seen in Figure 13 and Table 6. This suggests that Cercon is a stable material. Lava also revealed the same results before and after firing, however, the initial cores differed in colour which corresponds to the report by Denry and Kelly. (2008), about the concentration of shading liquid.

5.5 The distribution pattern between results and type of shading

The Pearson's chi-square test was conducted and in all instances the chi-square p- values were less than the value of significance of 0.05 implying that there was a significant relationship between results and the type of shading. This suggests that the type of shading influenced the result obtained in all materials. The unshaded specimens (white) remained the same shade and was unaffected by firing indicating that the unshaded specimens were the most stable, however, in a clinical laboratory setting, the zirconia material has to be shaded in order to obtain a superior aesthetic result as stated by Ishikawa-Nagai *et al.* (2013) and Kurtulmus-Yilmaz and Ulusoy (2014), regarding the study on peri-implant soft tissue colour. In this study, Zircon Zahn was the only product that gave 3 specimens with A2 colour after firing, which was the colour that was initially expected. The system that consistently produces the closest match to the desired shade should be considered in clinical practise.

5.6 One-way MANOVA analysis

5.6.1 Group

From the results it can be concluded that the multivariate effect of group is significant (Table 8). This suggests that the group type had an effect on the change in lightness and colour. A possible explanation for this finding would be that the control group did not undergo firing cycles whilst the experimental group was exposed to subsequent firings. The pairwise comparison indicates that there is a significant difference in the mean between ΔL and the group types (Table 10), where *p*=0.007. A possible explanation for this relationship would be that firing directly affects translucency and translucency has a direct effect on change in lightness.

Change in colour after subsequent repeated firings may also be accredited to the colour stability of metal oxides during firing which can affect the resulting colour of ceramic as mentioned by Spyropoulou *et al.* (2011). In the present study, an increase in the number of firings resulted in a greater change in the E* value of Lava and Zirkon Zahn specimens compared to Cercon. Several studies have suggested that certain metal oxides are not colour stable after they are exposed to firing temperatures, and pigment breakdown at firing temperatures have demonstrated colour changes of surface colorants (Denry and Kelly, 2008; Spyropoulou *et al.*, 2011). Research conducted by Mulla and Weiner (1991) investigated the colour change of orange and blue colorants as a result of firings. The study reported significant colour changes after the initial firing, but less distinct changes after successive firings. This differs to the results in the current study where distinct colour changes were mainly seen after successive firings.

The present *in vitro* study measured the colour changes of all ceramic specimens prepared with different all ceramic systems and fired different numbers of times. The results of this study support the hypothesis that colour differences would occur relative to the firing times.

Celik *et al.* (2008) reported that the final colour of an all-ceramic zirconia core is influenced by the firing procedure. This was further confirmed by Ozturk *et al.* (2008), who's study revealed significant changes in L*a*b*colour data with increased numbers of firings.

5.6.2 Sample Group Name

The mean difference is significant between the group types for each of ΔL and ΔE (p>0.05), except for Zirkon Zahn (*p*=0.073) and Lava (*p*=0.073) for ΔL (Table 12). This is comparable to the literature which also suggest that (ΔE) change in colour is more prevalent than (ΔL) change in lightness. Uludag *et al.* (2007), reported perceptual colour changes in L*a*b* colour values as the number of firings increased in a study investigating the influence of ceramic thickness and number of firings on the colour of ceramic systems. An increase in the number of firings resulted in a decrease in L* values and an increase in a* and b* colour values of In-Ceram and IPS Empress specimens with different dentin ceramic thicknesses.

5.6.3 Firing

A significant difference in ΔE (change in colour) was noticed from the first and third firings and the second and third firing *(p*<0.05) indicated by the pairwise comparison. The pairwise comparison also specified a significant difference in ΔL (change in lightness) from the first and third firings and the second and third firing *(p*<0.05). This suggests that there is a noticeable change in colour and lightness from the first to third firing but not from the first and second firings. These results were in line with that obtained by Uludag *et al*. (2007), concerning the L* coordinate, which also resulted in a significant (*p*<0.01) change between the second and third firing. The L* coordinate was reduced, thus the specimens became darker. A study by Pires-de-Souza (2009) found significant differences for all-ceramic specimens, amongst all sample groups, for a* and b* coordinates. After each firing there was an increase in both coordinates, which indicated that specimens became more reddish and yellowish with consequent firings.

This reduction in the current study was not significant between the groups fired 3 times and the groups fired 4 times. While studies by Seghi *et al.* (1989) and Barghi (1982), have confirmed a minimal effect of repeated firings on the colour of ceramic, O'Brien *et al.* (1990), reported significant differences between the colour of ceramic specimens that were fired 3 and 6 times. These restorations must undergo a minimum number of 2 firings (1 dentin and 1 enamel) in order to notice an effect as stated by O'Brien *et al.* (1990).

Other factors such as ceramic brand, shade and dentin porcelain thickness contribute extensively to colour changes as reported by Lee *et al*. (2007) and Ho-Jung *et al*. (2010).

5.6.4 Shaded and Unshaded

There was a significant difference seen in ΔE (change in colour) and ΔL (change in lightness) for both shaded and unshaded samples, where *p*<0.05 was suggested by the pairwise comparison. All significant values are highlighted in Table 20. This indicates that there is a noticeable change in colour and lightness in both shaded and unshaded samples (Table 20).

Only limited data could be found on the subject of shaded and unshaded zirconia core material only. Data consisted mainly on veneered cores. Aboushelib (2010) cited by Vichi *et al.* (2011)

reported that application of the required veneer ceramic over unshaded zirconia resulted in an accurate final shade whilst for shaded zirconia application of liner material or deep chroma dentin was necessary to obtain the desired result. It was further concluded that using pre-coloured zirconia did not offer any additional advantage over unshaded zirconia. In fact, a decrease in the strength of zirconia which had been coloured with shading liquid was noticed compared to zirconia that was left in its natural colour (Vichi *et al.,* 2011).

A study conducted by Kurtulmus-Yilmaz and Ulusoy (2014) reported a statistically significant difference in translucency between the different shaded specimens of control group when the veneered specimens were evaluated. This finding also indicated the likely effect of veneering ceramic on the overall translucency of the specimen.

Heffernan *et al.* (2002), reported that thickness of the core material had an effect on the translucency of all-ceramics. As a limitation of this study, all the core specimens were fabricated in a single thickness of 0.5 mm. Further studies are required to evaluate the stability of shaded zirconia core specimens in coping forms without the veneering ceramic.

CHAPTER SIX

CONCLUSION, LIMITATIONS AND RECOMMENDATIONS

6.1 Conclusion

The results of this study support the hypothesis that colour differences would occur relative to the number of firings for shaded zirconia. The insight gained in this study is imperative as it provides novel, accurate information on the stability of three all-ceramic zirconia systems that are available on the South African market. This may better assist dental technicians in their selection of such a system. Research has confirmed that all-ceramic restorations have a general success rate similar to those of metal ceramic crowns in terms of strength and aesthetics (Peumans *et al.*, 2000).

Various factors, however, could result in the aesthetic failure of an all-ceramic restoration, and many are associated with restoration fabrication procedures, such as the number of times the ceramic is fired which confirms the hypothesis of the present study. This factor could be the cause of the difference between the objective colour and the tangible colour reached in the final restoration. The extent of the colour difference of the restoration may be considered unsatisfactory and require replacement (Douglas and Steinhauer, 2007). The other contributing factor is the type of zirconia system selected which further emphasises that not all zirconia are alike. The scanning electron microscope analysis proved that there are variations in the microstructure of the different types of zirconia available.

6.2 Limitations and Recommendations

- Within the limitations of the present *in vitro* study of the various ceramic systems, it was determined that colour variations do occur after zirconia is exposed to numerous firings.
- In addition, differences in scanning electron microscope measurements were identified between the different all-ceramic systems. However, this study found minute changes in the grain size of all three dental zirconia systems after being exposed to numerous firing cycles up to 800^{o} C and an in-depth study on SEM is recommended. Optical properties should be further investigated before and after the application of veneering porcelain on ceramic core materials along with the human perception threshold.

- Further studies should be conducted on the effect of all-ceramic systems being exposed to numerous firing cycles with regards to translucency.
- It should be considered as a limitation of this study that specimens without veneering was used and is therefore recommended that future studies include copings with veneering ceramic.
- Inadequate information is available on the colouring liquids and their effect on core–ceramic veneer bonding (Aktas *et al.*, 2013). It is therefore suggested that future studies be conducted on the bond strength of veneering ceramic to zirconia cores.

CHAPTER SEVEN

REFERENCES

Aktas, G., Sahin, E., Vallittu, P., Özcan, M., Lassila, L. 2013. Effect of colouring greenstage zirconia on the adhesion of veneering ceramics with different thermal expansion coefficients. *International Journal of Oral Science,* 5:236–241.

Alghazzawi, T.F., Lemons, J., Liu, P., Essig, M.E., Janowski, G.M. 2012. Evaluation of the optical properties of CAD-CAM generated yttria-stabilized zirconia and glass-ceramic laminate veneers. *The Journal of Prosthetic Dentistry*. 107(5):300-308.

Anusavice, K.J. 2003. Phillip's Science of Dental Materials, 11th edition, St.Louis: WB Saunders Company.

Baldissara, P., Llukacej, A., Ciocca, L., Valandro, F.L. Scotti, R. 2010. Translucency of zirconia copings made with different CAD/ CAM systems. *The Journal of Prosthetic Dentistry,* 104:6-12.

Barghi, N. 1982. Color and glaze: effects of repeated firings. *The Journal of Prosthetic Dentistry,* 47:393-395.

Beck, N., Graef, F., Gerstbrein, O., and Karl, M. 2010. Sensitivity of transillumination for detecting microcracks in feldspathic and zirconia ceramic materials. *The Journal of Prosthetic Dentistry*, 104:301-305.

Behrens, A. 2008. 3M Espe Brochure. *Espertise Scientific Facts*. Seefeld, Germany.

Bona, A.D., Barrett, A.A., Rosa, V., Pinzetta, C. 2009. Visual and instrumental agreement in dental shade selection: Three distinct observer populations and shade matching protocols. *Dental Materials,* 29:276-281.

Burkinshaw, S.M. 2004. Colour in relation to dentistry. Fundamentals of colour science. *British Dental Journal*. 196:33-41.

Celik,G., Uludag, B., Usumez, A., Sahin, V., Ozturk, O. and Goktug, G. 2008. The effect of repeated firings on the colour of an all-ceramic system with two different veneering porcelain shades. *The Journal of ProstheticDentistry*, 99: 203-208.

Chen, Y.M., Smales, R.J., Yip, K.H., Sung, W.J. 2008. Translucency and biaxial flexural strength of four ceramic core materials. *Dental Materials*, 24:1506-1511.

Chu, S.J.,Trushkowsky, R.D., Paravina, R.D. 2010. Dental colour matching instruments and systems. Review of clinical and research aspects. *Journal of Dentistry,* 38s:e2-e16.

Conrad, H.J., Seong, W. and Pesun, I. J. 2007. Current ceramic materials and systems with clinical recommendations: a systematic review. *The Journal of Prosthetic Dentistry*, 98: 2007.

Corciolani, G., Vichi, A., Ferrari, M. 2009. Colour correspondence of a ceramic system in two different shade guides. *Journal of Dentistry,* 37:98-101.

Denry, I., Kelly, J.R. 2008. State of the art of zirconia for dental applications. *Dental Materials*, 24:299-307.

Douglas, R.D., Steinhauer, T.J., Wee AG. 2007. Intraoral determination of the tolerance of dentists for perceptibility and acceptability of shade mismatch. *The Journal of Prosthetic Dentistry*, 97:200-8.

Durand, J., Jacquot, B., Salehi, H., Fages, M., Margerit, J., Cuisinier, F.J.G. 2012. Confocal Raman microscopic analysis of the zirconia/feldspathic ceramic interface. *Dental Materials*, 28:661-671.

Edelhoff, D., Spiekermann, H., Rübben, A., Yildirim. M. 1999. Crown and bridge from high strength press ceramics. *Quintessence,* 50:177-189.

Geary, J.L., Kinirons, M.J. 1999.Colour perception of laboratory-fired samples of body-coloured ceramic. *Journal of Dentistry,* 27:145–148.

Hammad, I.A. Intrarater repeatability of shade selections with two shade guides. 2003. *The Journal of Prosthetic Dentistry*, 89:50-53.

Happe, A., Schulte-Mattler, V., Strassert, C., Naumann, M., Stimmelmayr, M., Zoller, J.E., Rothamel, D. 2013. In vitro colour changes of soft tissues caused by dyed fluorescent zirconia and nondyed, nonfluorescent zirconia in thin mucosa. *Quintessence,* 33:e1-e8.

Heffernan, M.J., Aquilino, S.A., Diaz-Arnold, A.M., Haselton, D.R., Stanford, C.M., Vargas MA. 2002. Relative translucency of six all-ceramic systems. Part I: core materials. *The Journal of Prosthetic Dentistry*, 88:4-9.

Ho-Jung, S., Woong-Chul, K., Sang-Ho, J., Young-Suk, S. J., Jin-Soo, A. 2010. Influence of dentin porcelain thickness on layered all-ceramic restoration colour. *Journal of Dentistry,* 10:106.

Ishikawa-Nagai, S., Wang, J., Seliger, A., Lin, J., Da Silva, J. 2013 Developing a custom dental porcelain shade system for computer colour matching. *Journal of Dentistry*, (Online). Available : http://dx.doi.org/10.1016/j.jdent.2012.12.006. Accessed on 26 June 2013.

Johnston, W.M., Kao, E.C. 1989. Assessment of appearance match by visual observation and clinical colorimetry. *Journal of Dental Restoration*, 68:819-822.

Jiang, L., Liao, Y., Wan, Q., Li, W. 2011. Effects of sintering temperature and particle size on the translucency of zirconium dioxide dental ceramic. *Journal of Mater Science: Material Medicine*, 22:2429–2435.

Jung, Y., Lee, J., Choi,Y., Ahn, J., Shin, S., Huh, J. 2010. A study on the in-vitro wear of the natural tooth structure by opposing zirconia or dental porcelain. *The Journal of Advanced Prosthodontics.* 2:111-5.

Kaya, G. 2013. Production and characterization of self-colored dental zirconia blocks. *Ceramics International*, 39:511-517.

Kim, M., Ahn, J., Kim, J., Kim, H., and Kim, W. 2013. Effects of the sintering conditions of dental zirconia ceramics on grain size and translucency. *The Journal of Advanced Prosthodontics*, 5:161-166.

Komine, F., Strub, J.R., Matsumura, H. 2012. Bonding between layering materials and zirconia frameworks. *Japanese Dental Science Review,* 48:153-161.

Kurtulmus-Yilmaz, S., Ulusoy, M. 2014. Comparison of the translucency of shaded zirconia all-ceramic systems. *Journal of Advanced Prosthodontics,* 6:415-422.

Lee, Y.K., Cha, H.S., Ahn, J.S., 2007. Layered colour of all-ceramic core and veneers. *The Journal of Prosthetic Dentistry*, 97:279-286.

Liu, M., Aquilino, S.A., Gratton, D.G., Ou, K., Lin,C. 2013. Relative translucency and surface roughness of four yttrium stabilised tetragonal zirconia polycrystalline – based dental restorations. *Journal of Exp. Clinical Medicine,* 5:22-24.

McLean, J.W., Kedge, M.I., Hubbard, J.R. 1976. The bonded alumina crown. 2. Construction using the twin foil technique. *Australian Dental Journal*, 21:262-268.

McLean, J.W. 1979. The science and art of dental ceramics, Volume 1. Chicago, IL: Quintessence Publishing Company.

Mulla, F.A., Weiner, S. 1991. Effects of temperature on color stability of porcelain stains. *The Journal of Prosthetic Dentistry*, 65:507-512.

O'Brien, W.J., Groh, C.L., Boenke, K.M. 1990. A new, small color difference equation for dental shades. *The Journal of Dental Restorations*, 69:1762-1764.

Øilo, M., Gjerdet, R. N. and Tvinnereim, H. M. 2007. The firing procedure influences properties of a zirconia core ceramic. *Academy of Dental Materials*, 24: 471-475.

Owen, C.P. 2012. On form and function in all-ceramic restorations. *South African Dental Journal*, 67:400-404.

Ozturk, O., Uludag. B., Usumez, A., Sahin, V., Celik, G. 2008. The effect of ceramic thickness and number of firings on the colour of two all-ceramic systems. *The Journal of Prosthetic Dentistry*, 100:99-106.

Paravina, R.D.,Powers, J.M., Fay, R.M. 2002. Colour comparison of two shade guides. *International Journal of Prosthodontics*,15:73-78.

Paravina, R.D. 2008. Performance assessment of dental shade guides. *The Journal of Prosthetic Dentistry*, 100: 99-106.

Pecho, O.E., Ghinea, R., Ionescu, A.M., Cardona, J., Paravina, R.D., Pe´rez, M. 2012. Color and translucency of zirconia ceramics, human dentine and bovine dentine. *Journal of Dentistry*. 40s:e34-e40.

Peumans, M., Van Meerbeek, B., Lambrechts, P., Vanherle, G. 2000. Porcelain veneers: a review of the literature. *Journal of Dentistry*, 28:163-77.

Pires-de-Souza, F.P., Casemiro, L.A., Garcia, L.R., Cruvinel, D. R. 2009. Color stability of dental ceramics submitted to artificial accelerated aging after repeated firings. *The Journal of Prosthetic Dentistry*, 101:13-18.

Raigrodski, A.J., 2004. Contemporary materials and technologies for all-ceramic fixed partial dentures: A review of the literature. *Journal of Prosthetic Dentistry,* 92:557-562.

Raptis, N.V., Michalakis, K.X., Hirayama H. 2006. Optical behaviour of current ceramic systems. *International Journal of Periodontics Restorative Dentistry,* 26:31-41.

Rosenblum, M.A., Schulman, A. 1997. A review of all-ceramic restorations. *The Journal of the American Dental Association,* 128:297-307.

Sahin, V., Uludag, B., Usumez, A., Ozkir, S.E. 2010. The effect of repeated firings on the colour of an alumina ceramic system with two different veneering porcelain shades. *The Journal of Prosthetic Dentistry,* 104:372-378

Sarafianou, A., Kamposiora, P., Papavasiliou, G., Goula, H. 2012. Matching repeatability and interdevice agreement of 2 intraoral spectrophotometers. *The Journal of Prosthetic Dentistry,* 107:178-185.

Schmidseder, J. 2000. Colour atlas of dental medicine: Aesthetic Dentistry. George Thieme Verlag; 2000; 5-6.

Seghi, R.R., Denry, I.L., Rosenstiel, S.F. 1995. Relative fracture toughness and hardness of new dental ceramics. *The Journal of Prosthetic Dentistry,* 74:145-150.

Seghi, R.R., Sorensen, J.A. 1995. Relative flexural strength of six new ceramic materials. *The International Journal of Prosthodontics*, 8:239-246.

Seghi, R.R., Hewlett, E.R., Kim, J. 1989. Visual and instrumental colorimetric assessments of small color differences on translucent dental porcelain. *Journal of Dental Restoration*, 68:1760-1764.

Siavikisa, G., Michael Behra, Van der Zelb, M.J.M., Feilzerb, A.J., Rosentritta, M. 2011. Influence of heat treatment and veneering on the storage modulus and surface of zirconia ceramic. *European Journal of Dentistry*, 5:191-198

Spink, L.S., Rungruanganuth, P., Megremis, S., Kelly, J.R. 2013. Comparison of an absolute and surrogate measure of relative translucency in dental ceramics. *Dental Materials*, 29:702-707.

Spyropoulou, P.E., Giroux, EC., Razzoog, M.E., Duff, R.E. 2011. Translucency of shaded zirconia core material. *The Journal of Prosthetic Dentistry*, 105:304-307.

Stevenson, B., and Ibbetson, R. 2010. The effect of the substructure on the colour of the samples/restorations veneered with ceramic: A literature review. *Journal of Dentistry*, 38:361-368

Suttor, D., Bunke, K., Hoescheler, K., Hauptmann, H., Hertlein, G. 2001. LAVA-the system for all-ceramic ZrO2 crown and bridge frameworks. *International Journal of Computerised Dentistry*. 4:195-206.

Tang, X., Nakamura, T., Usami, H., Wakabayashi, K., Yatani, H. 2012. Effects of multiple firings on the mechanical properties and microstructure of veneering ceramics for zirconia frameworks. *Journal of Dentistry*, 40:372-380.

Tinschert, J., Natt, G., Spiekermann, H. 2001. Current positioning of dental ceramics. *Dental Practise*, 12:293-309.

Uludag, B., Usumez, A., Sahin, V., Eser, K., Ercoban, E. 2007. The effect of ceramic thickness and number of firings on the color of ceramic systems: an in vitro study. *The Journal of Prosthetic Dentistry*, 97:25-31.

Vagkopolou, T., Koutayas, S.O., Koidis, P., Strub, J.R. 2009. Zirconia in dentistry: Part 1. Discovery of an upcoming bioceramic. *The European Journal of Esthetic Dentistry*, 4:2-23.

Vichi, A., Louca, C., Corciolani, G., Ferrari, M. 2011. Colour related to ceramic and zirconia restorations : A review. *Academy of Dental Materials*, 27:97-108.

Yamamoto, S., Hosoya, Y., Tsumura, N., Ogawa-Ochiai, K. 2012. Principal component analysis for dental shade colour. *Dental Materials*, 28:736-742.

Zhao, J. 2013. Bi-colored zirconia as dental restoration ceramics. *Ceramics International* (Online), Available: http://dx.doi.org/10.1016/j. ceramint.2013.05.036. Accessed 26 June 2013.

CHAPTER EIGHT

APPENDICES

Appendix A – Raw data collection

CONTROL GROUP

GROUP (a)

Shaded core	Unshaded core	Result	ΔE	ΔL
	a1	B1	12.5	8.7
	a1	B1	12.5	8.6
	a1	B1	14.1	10.2
a1s		B2	5.2	5.1
a1s		B2	5	2.7
a1s		B2	4.7	4.6
	a2	B1	13.3	9.3
	a2	B1	13.1	9.5
	a2	B1	12.9	9.2
a2s		B2	4	3.9
a2s		B2	4.3	4.3
a2s		B2	5.1	5
	a3	B1	13.9	9.9
	a3	B1	14.1	10
	a3	B1	12.7	8.7
a3s		B2	5.2	5.1
a3s		B2	4.5	4.4
a3s		B2	4.8	4.6

GROUP (b)

Shaded core	Unshaded core	Result	ΔE	ΔL
	b1	B1	13.3	6.9
	b1	B1	11.1	5.2
	b1	B1	12.2	5.2
b1s		A2	2.4	1.6
b1s		A2	2.4	0.7
b1s		A2	2.1	0.7
	b2	B1	13.1	7.7
	b2	B1	13.7	8.1
	b2	B1	13.9	8.3
b2s		A2	2.8	2.3
b2s		A2	2.3	1.5
b2s		A2	2.5	1
	b3	B1	13.5	7.7
	b3	B1	11.8	5.7
	b3	B1	13.6	7.9
b3s		A2	2.5	0.9
b3s		A2	2.6	1.2
b3s		A2	2.6	1

GROUP (c)

Shaded core	Unshaded core	Result	ΔE	ΔL
	c1	B1	12.3	6.4
	c1	B1	12.6	8.7
	c1	B1	12	6.8
c1s		A1	2.3	0.6
c1s		A1	3.9	1.4
c1s		B2	4	2.3
	c2	B1	11.3	5.2
	c2	B1	11.9	4.9
	c2	B1	12	6.8
c2s		B2	4.3	1.7
c2s		B2	4.1	1.5
c2s		A1	3.8	1.5
	c3	B1	12.8	8.8
	c3	B1	11	7
	c3	B1	13.9	9.1
c3s		A1	2.7	1.8
c3s		B2	4.4	1.7
c3s		B2	4.2	1.3

FIRST FIRING / SECOND FIRING / THIRD FIRING

GROUP (a)

Shaded core	Unshaded core	Result	ΔE	ΔL	Shaded core	Unshaded core	Result	ΔE	ΔL	Shaded core	Unshaded core	Result	ΔE	ΔL
	a1f1	B1	14	10.9		a2f2	B1	14	10.6		a3f3	B1	14.9	11.6
	a1f1	B1	14.2	11.1		a2f2	B1	14.4	11		a3f3	B1	14.6	11.1
	a1f1	B1	15.3	11.9		a2f2	B1	13.4	7.2		a3f3	B1	13.4	9.5
a1sf1		B2	5.6	5.4	a2sf2		B2	5.7	5.5	a3sf3		B2	6.5	6.3
a1sf1		B2	6.1	5.8	a2sf2		B2	5.8	5.6	a3sf3		B2	6.8	4.4
a1sf1		B2	5.8	5.6	a2sf2		B2	6.3	6.1	a3sf3		B2	6.1	5.9

GROUP (b)

Shaded core	Unshaded core	Result	ΔE	ΔL	Shaded core	Unshaded core	Result	ΔE	ΔL	Shaded core	Unshaded core	Result	ΔE	ΔL
	b1f1	B1	14	10.6		b2f2	B1	13.1	7.7		b3f3	B1	13.5	7.7
	b1f1	B1	12.2	9.2		b2f2	B1	13.7	8.1		b3f3	B1	11.8	5.7
	b1f1	B1	13.9	9.3		b2f2	B1	13.9	8.3		b3f3	B1	13.6	7.9
b1sf1		B2	2.4	0.6	b2sf2		A1	2.8	2.3	b3sf3		A2	2.5	0.9
b1sf1		A2	2.7	2.3	b2sf2		A2	2.3	1.5	b3sf3		B2	2.6	1.2
b1sf1		B2	2	0.5	b2sf2		B2	2.5	1	b3sf3		B2	2.6	1

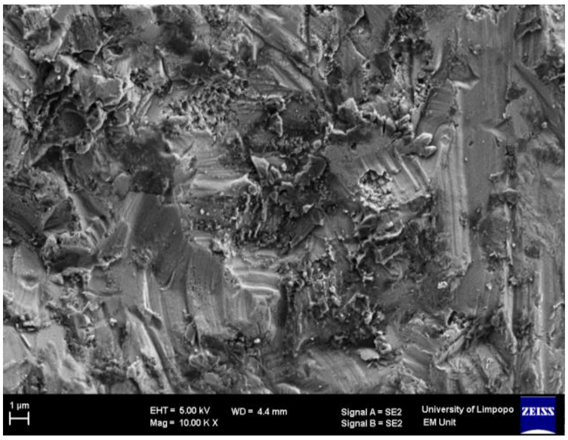
GROUP (c)

Shaded core	Unshaded core	Result	ΔE	ΔL	Shaded core	Unshaded core	Result	ΔE	ΔL	Shaded core	Unshaded core	Result	ΔE	ΔL
	c1f1	B1	10.9	4.2		c2f2	B1	12.2	6.1		c3f3	B1	14.1	9.4
	c1f1	B1	10.7	4.6		c2f2	B1	13.1	9.1		c3f3	B1	13.1	9.2
	c1f1	B1	10.5	3.9		c2f2	B1	12	7.5		c3f3	B1	11.8	7.6
c1sf1		A1	3.8	2.2	c2sf2		B2	4.7	3.6	c3sf3		A1	3.1	2.6
c1sf1		A1	3.1	2.7	c2sf2		B2	4.2	2.2	c3sf3		B2	12.4	10.9
c1sf1		B2	4.2	3.5	c2sf2		A1	3.8	2.1	c3sf3		B2	12.6	11

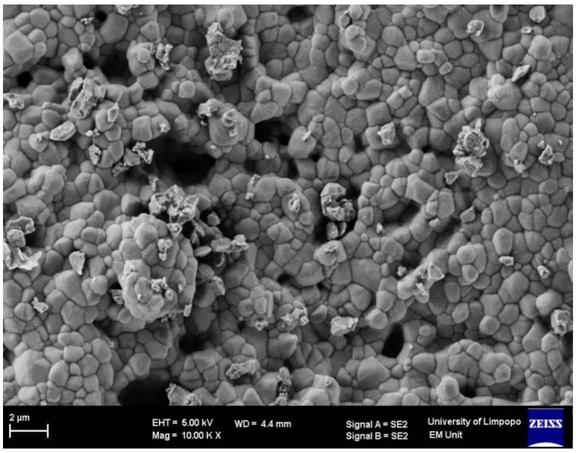
TRADE NAME	*LAVA unshaded*	*LAVA shaded*	*CERCON*	*CERCON SHAD*	*ZIRKON ZAH*	*ZIRKON ZAHN SHADED*
SAMPLE GROUP NAME	*c*	*c*	*a*	*a*	*b*	*b*
1ST FIRING	*c1*	*c1s*	*a1*	*a1s*	*b1*	*b1s*
2ND FIRING	*c2*	*c2s*	*a2*	*a2s*	*b2*	*b2s*
3RD FIRING	*c3*	*c3s*	*b3*	*b3s*	*b3*	*b3s*

Appendix B - Cercon SEM

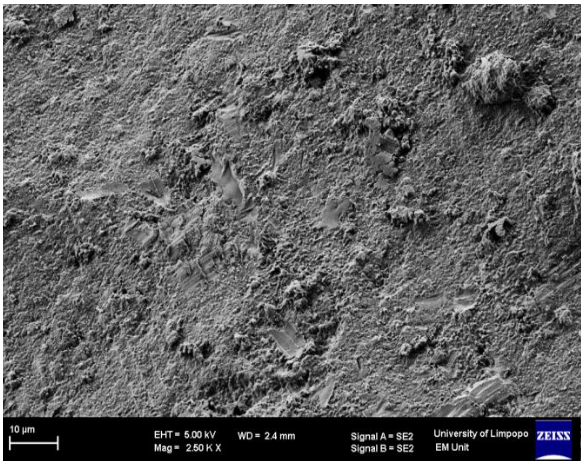
B1 - 1µm, Mag=10,00KX



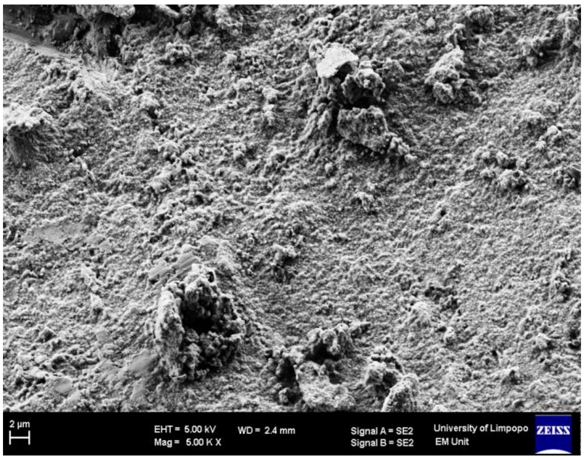
B2 - 2µm, Mag=10,00KX



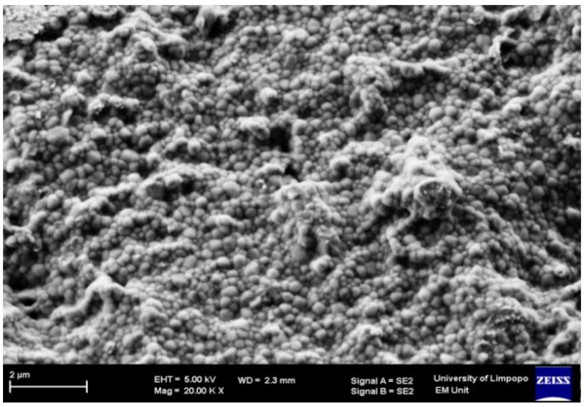
Appendix B3 - 10µm, Mag=2.50KX



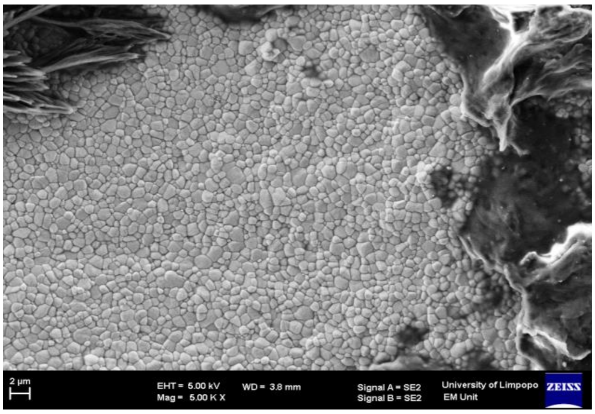
B4 1µm, Mag=5,00



B5 - 1µm, Mag=20,00KX

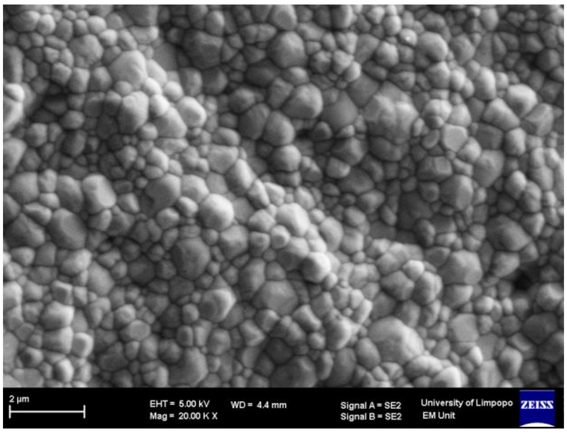


B6 - 1µm, Mag=5,00KX

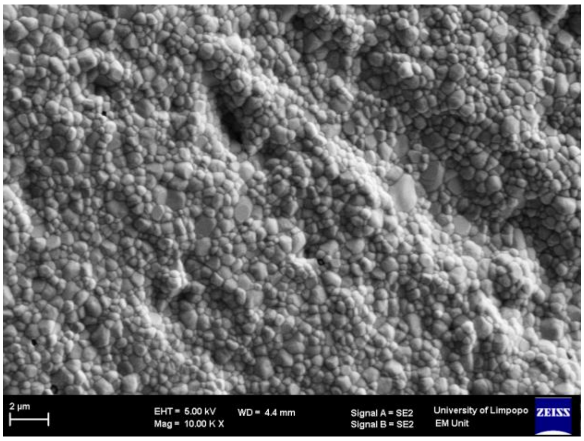


Appendix C – Zirkon Zahn SEM

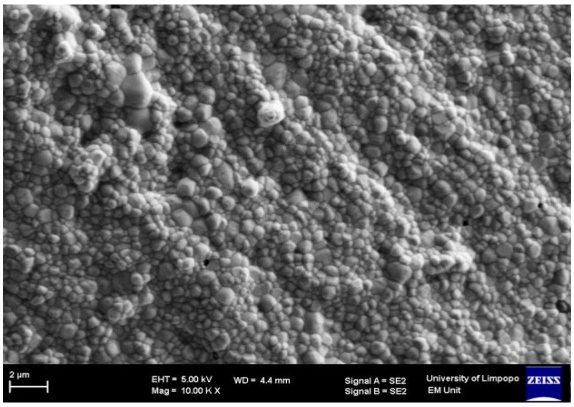
C1 - 1µm, Mag=10,00KX



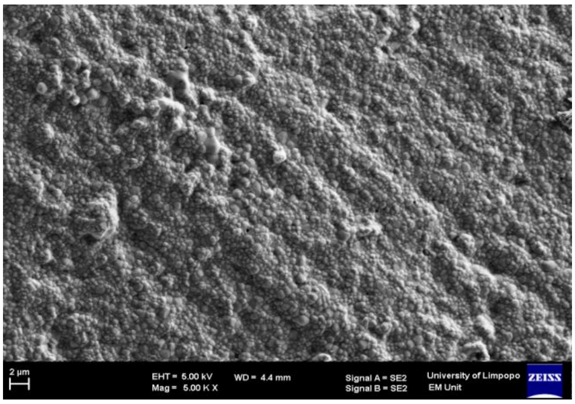
C2 - 2µm, Mag=10,00KX



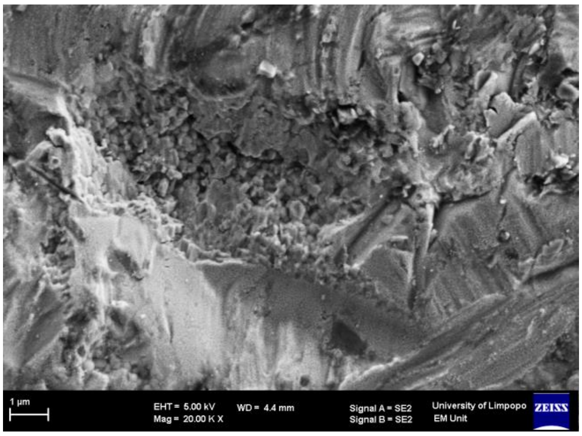
C3 - 2µm, Mag=10,00KX



C4 - 2µm, Mag=5,00KX



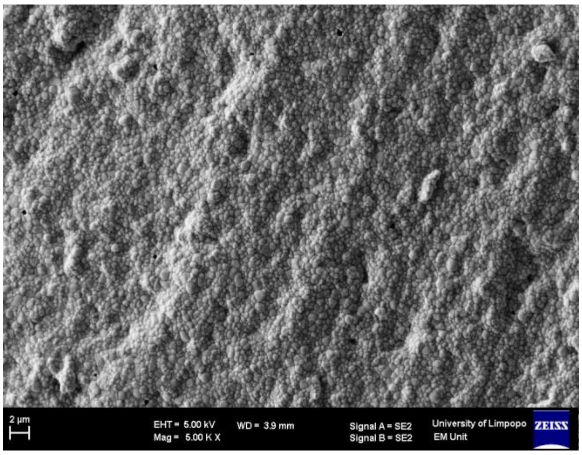
C5 - 1µm, Mag=20,00KX



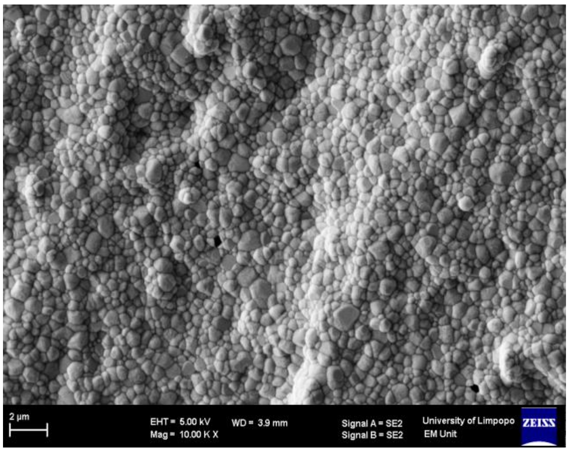
C6 - 1µm, Mag=20,00KX

Appendix D - Lava SEM

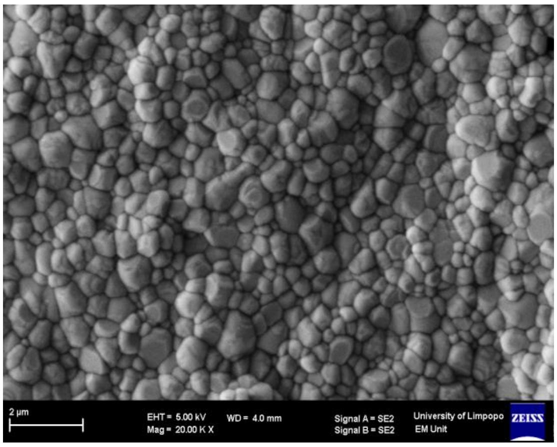
D1 - 2µm, Mag=5,00KX



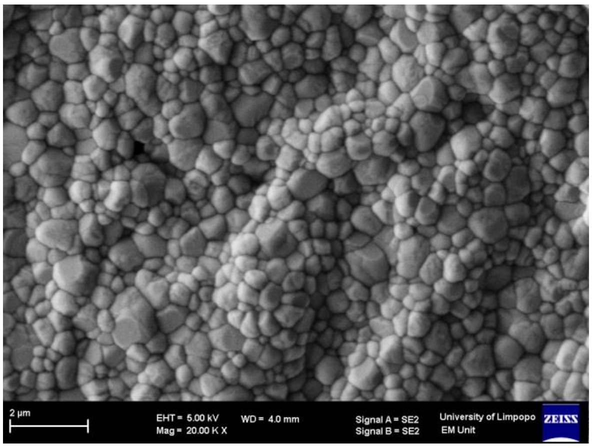
D2 - 2µm, Mag=10,00KX



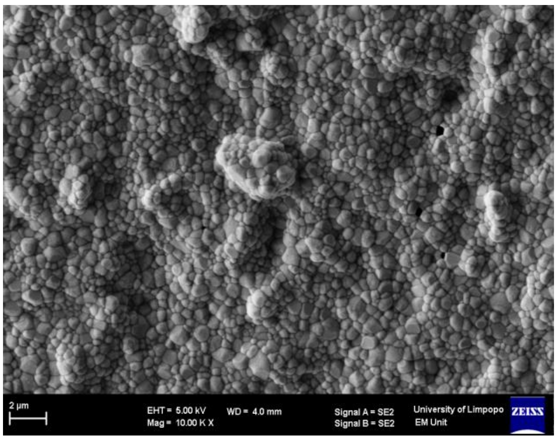
D3 - 2µm, Mag=20,00KX



D4 - 2µm, Mag=20,00KX



D5 - 2µm, Mag=10,00KX



D1 - 2µm, Mag=10,00KX