

**AN *IN-VITRO* STUDY ASSESSING THE COLOR STABILITY OF  
DIFFERENT PROVISIONAL CROWN AND BRIDGE  
RESTORATIVE MATERIALS**

A Mini-Thesis Submitted In Partial Fulfillment Of The Requirements For The Degree Of  
Master Of Science In Dental Sciences In Restorative Dentistry At The Faculty Of  
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**KEY WORDS**

Provisional crown and bridge restorative materials

Color stability

Staining agent

Polishing

Reflection spectrophotometer



## SUMMARY

Color stability of long-term provisional restorations for crown and bridge patients is a significant criterion in the selection of the type of provisional restorative material to be used. Discoloration of provisional restorations may result in patient dissatisfaction and an additional expense for their replacement. However, the evidence in the literature about the type of provisional restorative material that has enhanced color stability is conflicting. In addition, the need to devise mechanisms of improving the color of the stained provisional restorations is also apparent.

**Aim:** The aim of this study was to investigate the color stability of two provisional restorative materials used in crown and bridge cases upon exposure to different tea staining solutions and to evaluate the effectiveness of a polishing technique in removing the tea stains from the stained provisional restorative materials.

**Materials and Methods:** Two provisional restorative materials, Snap (polyethyl methacrylate) and Protemp 3 Garant (autopolymerized bis-acryl resin composite) were evaluated. Thirty specimens of each material were fabricated with dimensions of 10 mm in diameter and 2.5 mm thick. Each material was mixed and polymerized according to the manufacturer's instructions. All the specimens were stored in distilled water at room temperature for 24 hours after which baseline color measurements were recorded on a reflection spectrophotometer using CIE L\* a\* b\* relative to standard illuminant A against a white background. The 30 specimens of each material were randomly divided into 3 groups of ten specimens each. A group was stored in each of the two staining solutions (rooibos tea, Glen tea) and the third group was stored in distilled water and was used as the control group.

Color values of each specimen were taken after 1, 2, 3 and 4 weeks using the reflection spectrophotometer. At 4 weeks, all the specimens that had been immersed in the staining solutions were polished and a second reading was taken to determine the effect of polishing on the color of the stained specimens. Color changes at the specific time intervals were calculated for each specimen using the color difference formula:  $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$  where  $\Delta E^*$  represents the color difference and  $\Delta L^*$ ,  $\Delta a^*$ ,

$\Delta b^*$  represent the changes in lightness, changes in the red-green co-ordinate and changes in the yellow-blue co-ordinate respectively, after immersion in the various solutions.

**Results:** Results of the three factor analysis of variance (ANOVA) with repeated measures on time indicated that the effects of all three factors (materials, solutions and time), and all possible interactions among them, were statistically significant ( $P < .001$ ).

Pair-wise comparisons revealed that the type provisional restorative material and the solution significantly ( $P < .05$ ) affected the color stability at each immersion period, the interaction of the factors was also significant at each period of time.

Protemp showed marked discoloration for both teas compared to Snap specimens at all time intervals ( $P < .001$ ). Protemp specimens immersed in both teas showed a marked color change in the first week and thereafter (weeks 2, 3 and 4) the specimens only displayed slight color changes. At week four, Protemp had the highest color change for both rooibos tea and Glen tea ( $\Delta E > 15.0$ ). Snap was more color stable with a very slight color change ( $\Delta E < 2.0$ ) for both staining solutions. The Protemp specimens in rooibos tea showed the highest discoloration ( $\Delta E > 27.0$ ) at week four.

In distilled water, both materials showed very small changes in color ( $\Delta E < 1.5$ ) over the 4-week period.

Polishing of the discolored specimens caused a marked improvement in the color of the stained specimens ( $\Delta E > 15.0$ ). The polished specimens attained color values near to their baseline shades with a total color change of less than five units ( $\Delta E^* < 5.00$ ) compared to the baseline values. However, there were varying ranges of color values attained by the different specimens on polishing. There was no significant difference in the effect of polishing of the specimens for the two types of tea.

### **Conclusions:**

Within the limitations of this study, the type of provisional restorative materials, staining solutions and immersion times are significant factors that can affect color stability. Snap specimens were more color stable compared to Protemp specimens, with rooibos tea causing more discoloration of Protemp specimens compared to Glen tea. Discoloration caused by tea was removed or at least reduced by the polishing of the stained specimens.

## DECLARATION

I hereby declare that *An In-Vitro Study Assessing the Color Stability of Different Provisional Crown and Bridge Restorative materials* is my own work, that it has not been submitted before for any degree or examination in any University, and that all the sources I have used or quoted have been indicated and acknowledged by complete references.

Ndagire Barbara

Signed:.....



November 2006

## ACKNOWLEDGEMENT

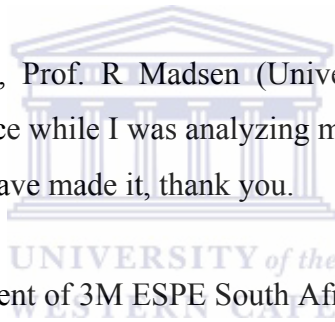
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Ndagire Barbara

## DEDICATION

This work is dedicated to:

- \* my parents for their constant support and sacrifice;
  
- \* my fiancé for his encouragement, support and sacrifice in ensuring that I pursue higher studies at the University of the Western Cape, far away from my home country; and to
  
- \* my supervisor whose guidance, encouragement, help and support made this project successful.



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# CHAPTER 1

## INTRODUCTION

Provisional crown and bridge restorations serve many purposes in prosthodontic treatment, including restoration of function, protection of the teeth and periodontal tissues, stabilisation of the occlusion and as a diagnostic evaluation prior to the fabrication of the final restoration as regards aesthetics (Burns *et al*, 2003 and Doray *et al*, 2003).

Although all these purposes are important, aesthetics of the provisional restoration is in many cases of prime importance to the patient especially in cases where the provisional restorations are going to be used for a long period of time and or are in the aesthetic zone (Koumjian *et al*, 1991; Burns *et al*, 2003; Doray *et al*, 2003; Guler<sup>a</sup> *et al*, 2005; Haselton *et al*, 2005).

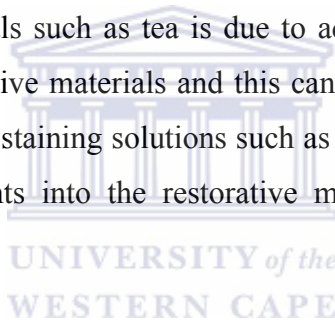
Discoloration of provisional restorations may result in patient dissatisfaction and an additional expense for their replacement (Sham *et al*, 2004; Ergun *et al*, 2005 and Haselton *et al*, 2005).

A number of materials are currently available for fabricating provisional fixed partial dentures. Most of these materials are composed of a methacrylate resin or a bis-acrylate composite resin. Each type of material has a unique chemical composition that imparts specific physical properties to the restoration (Haselton *et al*, 2004 and Haselton *et al*, 2005). Regardless of the specific chemistry, most provisional restorative materials are subject to sorption, a process of absorption and adsorption of liquids that occurs relative to the environmental conditions. As a result, color changes may occur over time when these provisional restorations are subjected to various staining agents (Burns *et al*, 2003 and Haselton *et al*, 2005).

A number of factors that include chemical and physical properties of the resin, incomplete polymerization, water sorption, chemical reactivity, diet (colorants in diet), oral hygiene, and surface roughness can affect color stability of these restorations (Scotti *et al*, 1997; Burns *et al*, 2003; Sham *et al*, 2004; Guler<sup>a</sup> *et al*, 2005).

It is still a contentious issue in research as to which type of material has the better color stability (polymethyl methacrylates, polyethyl methacrylates or bis-acryl composite resins) (Crispin and Caputo, 1979; Yannikakis *et al*, 1998; Doray *et al*, 2001; Sham *et al*, 2004; Guler<sup>a</sup> *et al*, 2005). Some researchers have concluded that almost all provisional restorative materials only have acceptable color stability for a short period of time and that they all discolor over a period of time if exposed to staining solutions (Kounjian *et al*, 1991; Scotti *et al*, 1997; Yannikakis *et al*, 1998).

Restorative materials are stained by various foods; the amount of staining caused by the different foodstuffs varies (Um and Ruyter, 1991; Bagheri *et al*, 2005). The degree of staining is also affected by the concentration of the staining agents and the amount of time the materials are exposed to the staining agents (Haselton *et al*, 2005). Um and Ruyter, (1991) contend that the mechanism of discoloration varies for the different foods. Discoloration by some materials such as tea is due to adsorption of the polar colorants onto the surface of the restorative materials and this can be removed by tooth brushing, whereas discoloration by other staining solutions such as coffee is due to both adsorption and absorption of the colorants into the restorative material (Um and Ruyter 1991; Bagheri *et al*, 2005).



These observations led to the research questions, as to which provisional restorative material has better color stability upon exposure to staining agents such as tea and the effectiveness of a polishing technique to remove the tea stains.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 PROVISIONAL RESTORATIVE MATERIALS

Provisional restorations are also referred to as interim or transitional restorations in the literature. However, the use of the term temporary is controversial and is considered inappropriate by many authors because provisional restorations serve many important functions, and temporary treatment may be interpreted as one of lesser importance or value (Zinner *et al*, 1989; Burns *et al*, 2003)

##### 2.1.1 Rationale for Provisional Treatment

Fixed prosthodontic treatment commonly relies on the indirect fabrication of the definitive prosthesis in the dental laboratory. As such patients must be provided with an interim restoration for the time interval between the initial tooth preparation and the placement of the definitive prosthesis and hence the primary indication for provisional treatment (Gratton and Aquilino, 2004).

Provisional restorations are also needed to maintain pulp vitality, occlusal position, and acceptable aesthetics during prosthodontic treatment. In addition, provisional restorations are used as diagnostic aids since they allow for the resolution of gingival inflammation and periapical lesions prior to the fabrication of the definitive prostheses (Doray *et al*, 2003; Guler<sup>a</sup> *et al*, 2005).

Provisional restorations may be needed for a long period of time in cases of extensive prosthodontic treatment such as treatment of patients with tooth wear, full mouth rehabilitations and in partially edentulous patients undergoing implant therapy (Galindo *et al*, 1998).

##### 2.1.2 Provisional Restorative Materials

Provisional restorative materials may be broadly classified into two categories namely custom fabricated materials and preformed materials (Burns *et al*, 2003).



- Preformed materials

Preformed provisional crowns or matrices usually consist of tooth-shaped shells of plastic, cellulose acetate, or metal. These preformed provisional crowns are commercially available in various tooth sizes and are usually selected for a particular tooth anatomy. Compared with custom fabricated restorations, temporisation with preformed materials is quick to perform but is more subject to abuse and inadequate treatment outcomes that can result in improper fit, contour, or occlusal contact for the provisional restoration (Christensen, 1996; Burns *et al*, 2003).

- Custom fabricated materials

Based on the method of polymerization, custom fabricated provisional restorative materials may be divided into the following categories (1) chemically activated autopolymerising acrylic resins; (2) heat activated acrylic resins; (3) light activated acrylic resins; (4) dual light and chemically activated acrylic resins, and (5) other alloys (Vahidi, 1987).

Acrylic resin materials are the most commonly used materials for the custom fabrication of provisional restorations. The several types of acrylic resin materials available for interim treatment include (1) polymethyl methacrylates, (2) polyethyl methacrylates, (3) other types or combinations of unfilled methacrylates and (4) composite resins (Burns *et al*, 2003; Gratton and Aquilino, 2004; Christensen, 2004).

#### *Methacrylate resins*

- *Polymethyl methacrylates*

Autopolymerizing polymethyl methacrylates (PMMA) remain the most frequently used material for the fabrication of interim restorations (Burns *et al*, 2003). The polymethyl methacrylates are the preferred material if the provisional restorations are to be made by the indirect method. However, in the direct method a major draw back of this type of material is the high intra-pulpal temperature rise associated with their polymerization that can lead to pulpal death (Burns *et al*, 2003; Gratton and Aquilino, 2004).

- *Ethyl methacrylate*

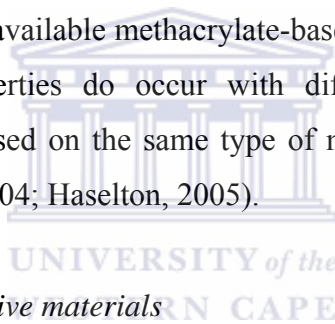
Ethyl methacrylates are suited for short-term use relative to the methyl methacrylates (Vahidi, 1987; Burns *et al*, 2003). They are a better selection for direct interim prosthesis fabrication since their advantages include lower exothermic reaction, less pungent odor, low polymerization shrinkage, good handling characteristics and good polishability (Burns *et al*, 2003).

- *Vinyl-ethyl and butyl methacrylates*

These two types of provisional restorative materials display comparable clinical behavior to the polyethyl methacrylates (Burns *et al*, 2003).

Generally, acrylic resins used for provisional restorations are brittle, but their great advantage is the ease with which they can be altered by additions and subtractions (Vahidi, 1987; Burns *et al*, 2003).

There are many commercially available methacrylate-based materials (Burns *et al*, 2003). However, variations in properties do occur with different commercially available materials even in materials based on the same type of methacrylate resin (Doray *et al*, 2001; Gratton and Aquilino, 2004; Haselton, 2005).



### *Composite provisional restorative materials*

Composite provisional restorative materials are chemically composed of a combination of two or more types of materials. Most of these materials use bis-acryl resin, a hydrophobic material that is similar to the bisphenol-A-glycidyl methacrylate (bis-GMA) present in composite restorative materials. These composite provisional restorative materials are available as autopolymerised, or dual-(auto/visible light) polymerized, or visible light-polymerised forms (Burns *et al*, 2003).

- *Autopolymerized composites*

Most of the composite provisional restorative materials available have an auto-mix delivery system similar to the polyvinylsiloxane impression materials. This makes them quick and easy to use.

- *Visible light-polymerized resins*

The visible light polymerized (VLC) materials require the addition of urethane dimethacrylate, a resin whose polymerisation is catalyzed by visible light energy and a camphoroquinone/ amine photo initiator.

- *Dual-polymerizing composite materials*

Dual-polymerizing composite materials generally incorporate both chemically polymerized bis-acryl and light-polymerized urethane dimethacrylate resins in variable product-specific combinations (Burns *et al*, 2003).

Like the methacrylates there are many commercially available composite based provisional restorative materials on the market with slightly differing properties depending on the chemical composition (Doray *et al*, 2001; Burns *et al*, 2003; Gratton and Aquilino, 2004; Haselton *et al*, 2005).

### **2.1.3 Requirements for Provisional Restorative Materials and Criteria for their Selection**

The desired physical properties of provisional restorative materials include minimal temperature change during polymerization, high surface hardness, good marginal fit, wear resistant, transverse strength, transverse repair strength, high polishability, color stability and stain resistance (Wang *et al*, 1989; Burns *et al*, 2003; Gratton and Aquilino, 2004).

Presently there is no ideal provisional restorative material suitable for all clinical conditions and that meets all the requirements; however, there are many materials that have been used successfully for this purpose. Material selection is often based on the strength and weakness of a given material relative to the clinical mandates for specific treatment (Wang *et al*, 1989; Burns *et al*, 2003; Gratton and Aquilino, 2004). For example in the esthetic zone or if the provisional restorations are to be used for a long time, color stability is a major criterion in the selection of the material to be used as the interim restorative material (Burns *et al*, 2003; Doray *et al*, 2003; Guler<sup>a</sup> *et al*, 2005; Koumjian *et al*, 1991; Haselton *et al*, 2005).

## 2.2 COLOR STABILITY

### 2.2.1 Color Stability of Provisional Restorations

In esthetically critical areas, it is desirable for provisional restorations to provide an initial accurate color shade match and to remain color stable over the course of provisional treatment (Gratton and Aquilino, 2004; Doray *et al*, 2003; Guler<sup>a</sup> *et al*, 2005; Koumjian *et al*, 1991; Haselton *et al*, 2005).

Most provisional restorative materials are subject to sorption, a process of absorption and adsorption of liquids that occurs relative to the environmental conditions. As a result, provisional restorations may discolor over time when they come into contact with pigmented solutions such as coffee, tea or wine (Haselton *et al*, 2005 and Burns *et al*, 2003).

### 2.2.2 Factors Affecting Color Stability

Color stability of these provisional restorations can be affected by a number of factors including the chemical and physical properties of the resin (Scotti *et al*, 1997), incomplete polymerization, water sorption, porosity, chemical reactivity, diet (colorants in diet), oral hygiene and surface roughness (Crispin and Caputo, 1979).

In an attempt to improve on the color stability of provisional restorations, research has been conducted on the factors affecting color stability. Recommendations include the use of a particular type of material as it has a better color stability *in vitro* (Sham *et al*, 2004 and Scotti *et al*, 1997), while others have advocated the use of different polishing techniques of provisional restorations to achieve a smooth surface with a resultant improvement in color stability (Sen *et al*, 2002; Guler<sup>a</sup> *et al*, 2005).

- Chemical and Physical Properties of Resins

It is still a contentious issue as to which type of material has a better color stability over a period of time. Several studies have indicated that the polymethyl methacrylates have better color stability compared to other types of materials (Guler<sup>a</sup> *et al*, 2005; Yannikakis *et al*, 1998; Crispin and Caputo, 1979). Guler<sup>a</sup> *et al*, (2005) in an *in vitro* study concluded that the methyl methacrylate-based material tested was more color stable compared to the chemically polymerized bis-acryl composite and the light polymerized composite

provisional restorative materials tested. Yannikakis *et al*, (1998) also found that the composite-based materials, especially the light-curing composite materials, were the least color stable in their study. Crispin and Caputo, (1979) also found that the methyl methacrylate materials exhibited the least darkening, followed by the ethyl methacrylate and the vinyl-ethyl methacrylate materials.

However, research (Doray *et al*, 1997; Doray *et al*, 2001 and Sham *et al*, 2004) has also demonstrated that there are resin composites of similar or better color stability compared to the polymethyl methacrylates. Sham *et al*, (2004) in an *in vitro* study concluded that the bis-acryl resins (Luxatemp and Integrity) demonstrated acceptable color stability and were the most color-stable provisional prosthodontic materials tested compared to the methyl and ethyl methacrylate-based resins. Doray *et al*, (2001) and Doray *et al*, (1997) in their studies demonstrated that some resin composites (Integrity, Luxatemp and Protemp Garant) were as color stable as acrylic resins tested following exposure of materials to *in vitro* accelerated aging conditions.

Other researchers have found all the materials to have poor color stability; Yannikakis *et al*, (1998) observed that after immersion in coffee or tea solutions for seven days, all provisional restorative materials showed observable color changes. Scotti *et al*, (1997) in an *in vitro* study investigating the color stability of acrylic resins for provisional restorations using computerized spectrophotometry found that only the Cold Pac resin was color stable in all staining solutions, while all the other materials showed color changes due to the different staining solutions. Yaman *et al*, (1989) also found poor color stability of all provisional restorations assessed in an *in vitro* study.

From the above studies on color stability of the different provisional restorative materials, it may be concluded that all provisional restorative materials have unpredictable color stability over an extended period of time. The purpose of this study was to investigate the color stability of the two groups of provisional restorative materials namely Snap (Parkell, USA) a polyethyl methacrylate and Protemp 3 Garant (ESPE, Germany) a bis-

acryl composite resins over a period of four weeks when immersed in different tea solutions.

- Effect of different polishing techniques

Different researchers have advocated the use of one or a combination of techniques to achieve an improvement in color stability. Sen *et al*, (2002) in an *in vitro* study found that diamond-polishing paste produced smoother surfaces for both bis-acrylic composites and the methacrylate-based provisional restorative materials. In another study, Guler<sup>a</sup> *et al*, (2005) found that better color stability was achieved if the provisional restorative materials were initially polished with pumice and then followed by polishing with diamond polishing paste.

However, in another study Haselton *et al*, (2004) found that there were significant differences in the surface roughness of the provisional restorative materials even when they were polished under the same conditions. In addition previous studies on finishing of resin-based restorations showed that very smooth surfaces can be obtained when restorations were allowed to set in contact with matrix strips (Heath and Wilson, 1976; McLundie and Murray, 1972; Sen *et al*, 2002)

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## **2.3 STAINING OF RESTORATIONS**

### **2.3.1 Staining of Aesthetic Restorative Materials**

Aesthetic restorative materials are stained by various foods (Um and Ruyter, 1991; Bagheri *et al*, 2005, Guler<sup>b</sup> *et al*, 2005). However, the amount of the staining varies with the different food materials (Bagheri *et al*, 2005). In an *in vitro* study to determine the effect of various food staining materials on aesthetic restorative materials Bagheri *et al*, (2005) found that coffee, red wine and tea caused more staining of the restorative materials tested compared to any other food simulating solutions such as soy sauce and cola.

Several researchers have used solutions of coffee or tea, or artificial saliva and coffee or tea solutions in *in-vitro* studies to determine the color stability of provisional restorative materials (Crispin and Caputo, 1979; Koumjian *et al*, 1991; Scotti *et al*, 1997; Yannikakis

*et al*, 1998; Guler<sup>a</sup> *et al*, 2005). Many studies have shown that the color change of provisional restorative materials varies with the staining agent, concentration of the staining agent and the duration of exposure to the staining agent (Crispin and Caputo, 1979; Koumjian *et al*, 1991; Scotti *et al*, 1997; Yannikakis *et al*, 1998).

### 2.3.2 Staining Techniques

Various techniques have been employed to elicit color changes in *in-vitro* experiments. These include exposure to accelerated aging conditions and immersion in staining solutions.

- Accelerated aging

Accelerated aging conditions have been used to study the effect of a controlled set of conditions on the color stability of provisional crown and bridge resins. Doray *et al*, (2001) and Doray *et al*, (1997) demonstrated that clinically perceptible and unacceptable color changes occur in some tested resins after accelerated aging. Although accelerated aging conditions are not able to replicate the complex *in vivo* environment, they allow researchers to assess the response of the provisional resins to simulated conditions with one variable factor thus considering the potential of these provisional resins for clinical color stability over time (Doray *et al*, 2001).

- Immersion in staining solutions

Other investigators have used immersion solutions such as artificial saliva, coffee, tea, grape juice, chlorohexidine and artificial saliva and tea or coffee solutions over measured time intervals to compare their effects on color stability of provisional and direct restorative resins (Crispin and Caputo, 1979; Um and Ruyter, 1991; Yannikakis *et al*, 1998; Haselton *et al*, 2005; Bagheri *et al*, 2005). The type of immersion solution used affects the degree of color change of the restorative material tested, with coffee and tea often contributing to the most significant staining of the restorative materials tested (Bagheri *et al*, 2005).



Based on the evidence in the literature (Bagheri *et al*, 2005 and Um and Ruyter, 1991), different tea solutions (Green tea and rooibos tea) were used in this study as the immersion solutions to assess the color stability of the provisional restorative materials tested.

### **2.3.3 Mechanism of Staining**

Um and Ruyter, (1991) and Bagheri *et al*, (2005) suggested that discoloration of the esthetic restorative materials may be due to a yellow colorant found in most food materials. Absence of the yellow colorant may explain why some materials like cola do not cause much discoloration (Bagheri *et al*, 2005). However the polarity of the yellow colorant present in the different materials varies and so does the mechanism of discoloration (Um and Ruyter, 1991).

Um and Ruyter, (1991) further explained that tea and coffee had yellow colorants with different polarities that have different mechanisms of staining. Discoloration by tea is due to adsorption of the polar colorant onto the surface of the restorative materials, this can be removed by tooth brushing, whereas discoloration by coffee is due to both adsorption and absorption of the colorants into the restorative material (Um and Ruyter 1991; Bagheri *et al*, 2005).

Based on the evidence in the literature (Um and Ruyter, 1991) this study tried to determine the efficiency of a polishing technique to remove the different tea stains, knowing that some tea solutions stained provisional restorative materials by adsorption of colorants onto the surface, thus polishing of the stained restorations would assist in improving the color of the stained restorations.

## **2.4 COLOR MEASUREMENT**

### **2.4.1 Factors that Affect Color Measurement of a Material**

Color measurement of a specimen depends not only on the actual color of the surface but also on several factors that include lighting conditions under which the surface color is measured, thickness of the material, and smoothness of the specimen (Koishi *et al*, 2001; Guler<sup>a and b</sup> *et al*, 2005).

In this study, a standard illuminant A against a white background was used to calibrate the readings during color measurements and the provisional restorative material



specimens prepared were 2.5 mm thick. However, since color changes were being measured, the choice of the illuminant was not important (Guler<sup>a and b</sup> *et al*, 2005)

#### **2.4.2 Instruments Used for Color Determination**

Color can be evaluated visually and by instrumental techniques (Um and Ruyter, 1991; Paul *et al*, 2004). However, visual evaluation of color is highly subjective. General variables such as external light conditions, experience, age, fatigue of the human eye and physiologic variables such as color blindness can lead to inconsistencies of color measurements made by this method (Paul *et al*, 2004). Instrument measurements eliminate the subjective interpretation of visual color comparison, thus spectrophotometers and colorimeters have been recommended to measure color stability of dental materials during these experiments (Um and Ruyter, 1991; Paul *et al*, 2004; Guler<sup>a</sup> *et al*, 2005)

- Instrumental techniques

In dentistry, shade guides, colorimeters and spectrophotometers are used for color measurements. Though shade guides are frequently used in color determination for indirect restorations, commercially available shade guides vary greatly and lack standardization (Paul *et al*, 2004). The use of the colorimeter and spectrophotometer is considered to increase the accuracy and reproducibility in color determination (Um and Ruyter, 1991; Paul *et al*, 2004).

Colorimeters and spectrophotometers give control over external light conditions, and the photo-optical measurement allows quantification of color using CIE (Commission Internationale de l'Eclairage) L\*a\*b\* coordinates (where L\* is the lightness, a\* is the chroma along the red-green axis and b\* is the chroma along the yellow-blue axis). Based on such CIE L\*a\*b\* parameters, data on the color obtained allows for an objective mathematical comparison between two shades. Thus, the CIE Lab system for measuring chromacity is well suited for determination of small color differences that may occur (Khokhar *et al*, 1991; Paul *et al*, 2004).

Based on the above advantages of the CIE Lab system for measuring chromacity a reflection spectrophotometer (Konica Minolta Sensing Inc., Japan) was used for the color determination in this study.

### **2.4.3 Using a Reflection Spectrophotometer**

A spectrophotometer measures color values in three different coordinates, L, a and b (CIE L\* a\* b\* color parameters); where L\* represents values in the lightness (black to white), a\* represents values in the red-green coordinate, and b\* represents values in the yellow-blue coordinate. All the values are absolute numbers with the following explanation. (1) the L\* coordinate (range 0-100) measures the quantity of white-black: the greater the L value, the whiter the sample (0= black, 100= white); (2) the a\* coordinate measures the color along the red-green axis: a positive a\* value refers to the amount of red in the sample, a negative a\* value refers to the amount of green; (3) the b\* coordinate measures the color along the yellow-blue axis: a positive b\* value is yellow, a negative b\* value is blue (Scotti *et al*, (1997) and Haselton *et al*, (2005)- quote CIE Commission Internationale de L'Eclairage, 1978).

### **2.4.4 Determination of Color Changes**

The color changes at the specific time intervals can be calculated for all three coordinates ( $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ) with the total color change ( $\Delta E^*$ ) being calculated using the color difference formula:  $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{\frac{1}{2}}$ , where  $\Delta E^*$  represents the total color difference and  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  represent the changes in lightness, red-green coordinate, and yellow-blue coordinate respectively between the two periods of measurement (Scotti *et al*, 1997 and Haselton *et al*, 2005).

## **2.5 REVIEW OF TESTED MATERIALS**

### **2.5.1 Provisional Restorative Materials Tested**

The provisional restorative materials tested were Snap a polyethyl methacrylate and Protemp™ 3 Garant™ a bis-acryl composite resin. The scientific information of each material tested was obtained from the product leaflets or the manufacturer's website. The details for the materials used are summarized in Table 2.1

**Table 2:1 Materials tested**

<b>Commercial name</b>	<b>Composition</b>	<b>Polymerisation</b>	<b>Shade</b>	<b>Manufacturer</b>
Snap	Polyethyl methacrylate	Autopolymerization	61	Parkell, Farmingdale, N.Y. USA
Protemp Garant	Bis-acryl composite resin	Autopolymerization	A2	ESPE, Seefeld, Germany

### 2.5.1.1 Protemp™ 3 Garant™ (<http://www.3mespe.com>, 2006)

- Composition

Protemp™ 3 Garant™ is a bis-acryl composite resin-based provisional restorative material manufactured by 3M ESPE Seefeld, Germany. The bis-acryl resin is a hydrophobic material that is similar to the bis-GMA found in restorative composite resins. Protemp™ 3 Garant™ has been available commercially in South Africa since 2001; the product is an improvement of the previous composite based provisional restorative material (Protemp Garant) produced by the same company. The material is available in several shades A1, A3, B0.5, B3 and a bleach shade.

- Indications

- Fabrication of temporary crowns, bridges, inlays, onlays and veneers
- Crown lining material for 3M™ ESPE™ prefabricated temporary crowns

- Product Composition

This is a two-component base/catalyst paste system that is available in a double chamber cartridge. The mixing ratio of the base paste to the catalyst paste is 10:1. The base paste contains dimethacrylate, strontium glass powder, silica powder, di-acrylates, stabilizers, synthetic resins, pigments and dyes. The catalyst paste contains a softener, strontium glass powder, initiators and pigments.

The inorganic fillers are incorporated into the organic matrix by a chemical process when the fillers are silanized; this produces a mechanically stable composite material that is wear-resistant, radiopaque and polishable. In addition, polymerization shrinkage is

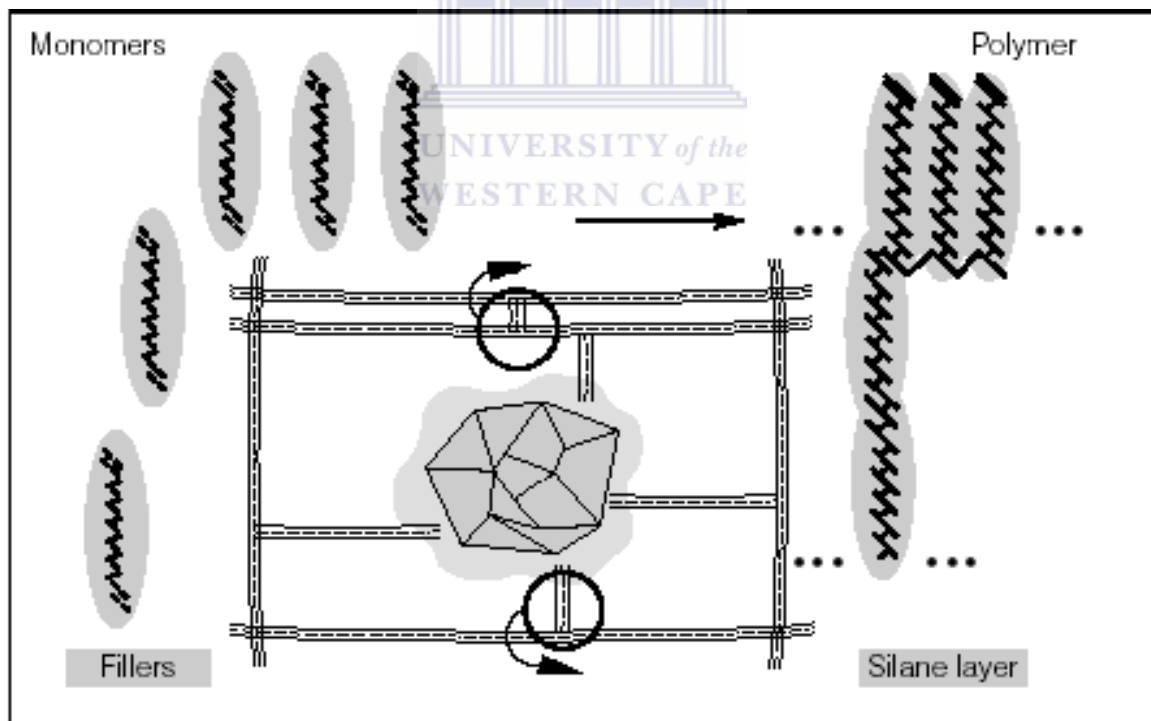
greatly reduced in comparison to the filler-free materials and the temporary restorations made using this material have a good precision of fit (<http://www.3mespe.com>, 2006).

- Polymerisation

Protemp™ 3 Garant™ is a self-cured (autopolymerised) composite resin based provisional restorative material. Polymerisation occurs when the initiator components come together during mixing of the base and the catalyst pastes. An oxidation-reduction (redox) reaction occurs.

Bis-acrylate composites have a bifunctional monomer, that is, they contain two double bonds capable of reacting. The multiple functionality of these monomers in bis-acrylate composites ensures the formation of a three-dimensional network, with the structure fixed by chemical bonds as shown in figure 2.1.

**Figure 2.1 Formation of a polymer network** (<http://www.3mespe.com>, 2006)



- Advantages of Protemp™ 3 Garant™ (According to the manufacturer 3M, ESPE, Seefeld, Germany)
  - Comparatively low setting temperature and polymerization shrinkage resulting in a good precision fit
  - Good handling properties-cartridge dispensed and automixed
  - Compatible with all impression materials, suitable for laboratory-made templates or strip crowns for single tooth restorations.
  - Anatomical remodeling and individualization in terms of shape and color can be done
  - Supplementation or repair is possible using other flowable composites or with the same material
  - Contains finer filler particles resulting in good polishability
  - Permits limited deflection that is higher than for standard composites, but does not mimic the behavior of MMA/PMMA materials, that can undergo great deflection but do not recover afterwards. At the same time high flexural strength and high flexural resistance (characteristic value: elastic modulus) are required to relieve the stress on the periodontia
  - Manufacturer also claims that the material has good color stability
  
- Handling characteristics: dispensing, mixing, and application
  - Dispensed and auto-mixed from the Garant 2 dispenser, material is easily loaded without causing voids.
  - The material attains a hard-elastic consistency approximately 1 min 35 sec after the onset of mixing.
  - The material and the impression or vacuum-formed template must be removed from the mouth within 2 minutes 30 seconds after the onset of mixing.
  
- Finishing
  - May be finished once the material is completely cured (not earlier than 5 minutes after the onset of mixing) using fine carbide burs and polished if desired.

- The oxygen-inhibited layer on the surface of the restoration can be removed with organic solvents such as ethanol.

#### **2.5.1.2 Snap** ([www.prestigedental.co.uk/parkell](http://www.prestigedental.co.uk/parkell), 2006 and <http://www.3mespe.com>, 2006)

Snap is a polyethyl methacrylate made by Parkell, Farmingdale, N.Y. USA. Like the other ethyl methacrylates, it is suited for short-term provisional restorations fabricated at the chair-side. Snap is available in different shades 59, 61, 62, 65, 69, 77, 81 and as a clear version.

- **Composition and Dispensing**

Snap consists of a powder and liquid (monomer). The powder is a polyethyl methacrylate while the liquid is an ethyl methacrylate

- **Polymerization:** This is a self-curing material where polymerisation occurs once the powder and liquid are mixed. An oxidation-reduction reaction occurs producing free radicals ( $R\bullet$ ) that are capable of attacking the double bonds of the acrylate group and in itself generating a radical. This process is called a chain initiation reaction shown in figure 2.2

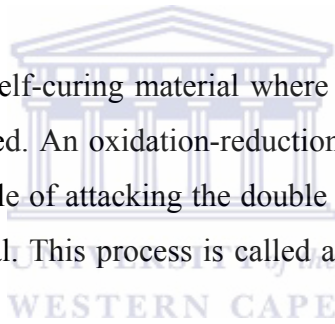
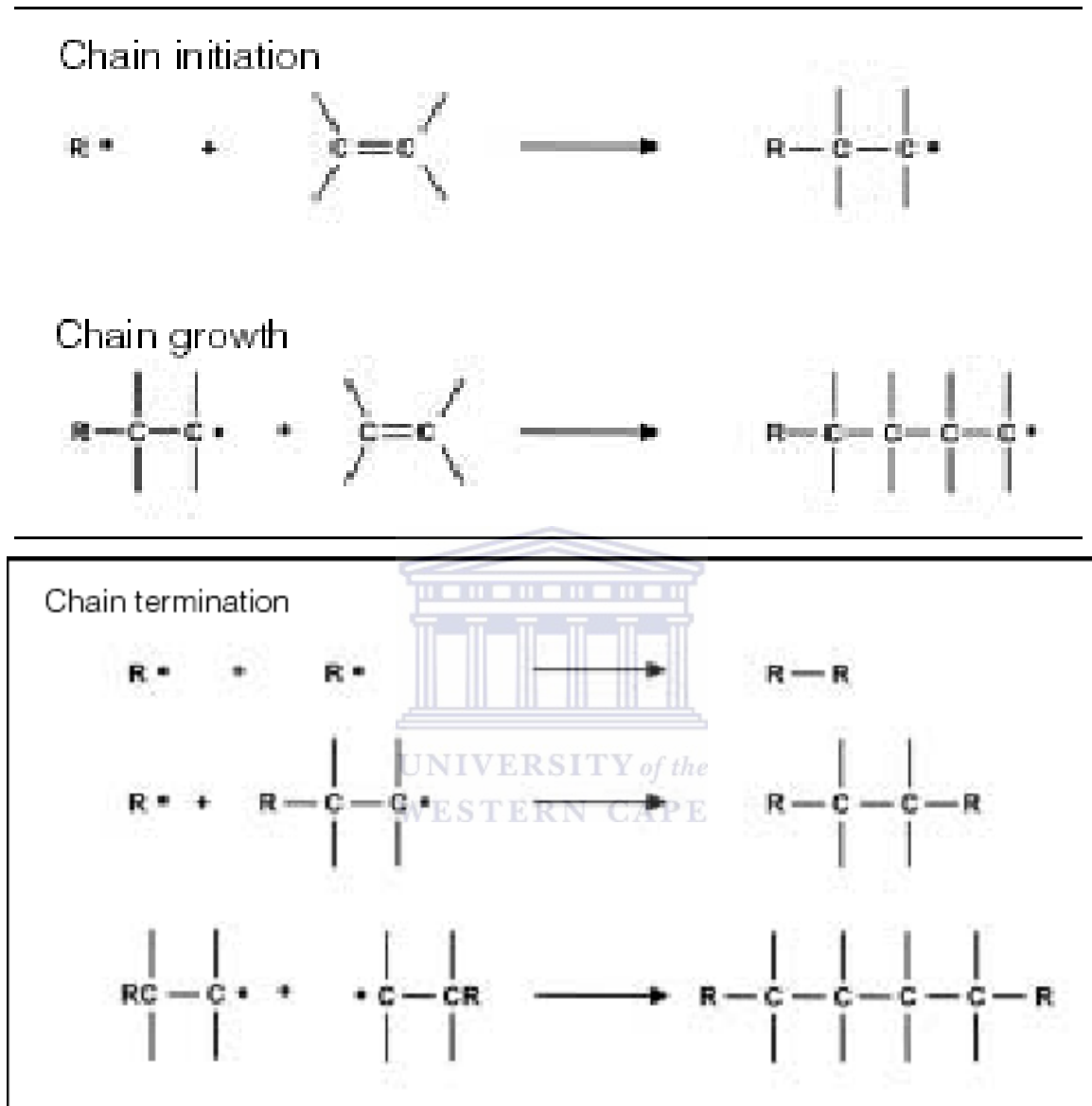


Figure 2:2 Steps involved in the polymerization reaction (chain initiation, chain growth and chain termination) (<http://www.3mespe.com>, 2006).



The chain growth reaction will continue as long as the free radicals encounter a double bond. As the chain is extended—the process represents actual polymerization; molecules of ever-increasing size are formed. Only when two radicals directly encounter each other are they recombined and the reaction finishes in a chain termination reaction as shown in figure 2.2. This stops further growth of the chain.

The polymer matrix formed consists of monofunctional low-molecular monomers, thus only linear chainlike polymers can be formed. Three-dimensional interlacing is only

possible through physical looping of the individual polymer strands, and the resulting framework is not very stable.

- Advantages
  - Low cost
  - Compared to the polymethyl methacrylates, snap (a poly ethyl methacrylate) gives off less heat during the polymerisation reaction and undergoes less shrinkage on polymerisation
  - It has a less pungent odor compared to polymethyl methacrylates.
  - Has an extended working time
  - Can be repaired by adding to it.
  
- Handling characteristics: Dispensing, Mixing, and application
  - The mixing ratio of powder to liquid is 3:1.
  - Mixing time is 2-3 minutes from the start of the mix.
  - The rubbery stage is of 3-5 minutes duration.
  - Final hardening is 6-7 minutes from the start of the mix.
  - Final trimming is done with acrylic burs and the provisional restoration may be polished with pumice.

### **2.5.2 Review of Staining Solutions: Tea Solutions**

Tea is the most consumed beverage aside from water in the entire world. It has been estimated that over one-half of the world's population drinks tea (Cheng, 2006; <http://www.tea@everything2-com.html>, 2006). There are many different types of tea available that are consumed worldwide. Choice of the type of tea one takes depends not only on the final taste of the tea but also on the presumed medical benefits of the type of the tea consumed (Cheng, 2006; Strand Tea Company, 2006).

In recent years many studies have shown that various types of tea have a wide range of physiological, biochemical and pharmacological effects due to the properties of their constituents (Kunishiro *et al*, 2001; Uličná *et al*, 2003; Strand Tea Company, 2006). Tea is presumed to have positive medical benefits in many illnesses that include



atherosclerosis, coronary heart disease, hypertension, diabetes, metabolic syndrome, obesity, cancer, HIV and allergies (Cheng, 2006; Marnewick *et al*, 2000; Marnewick *et al*, 2005; Strand Tea Company, 2006).

### 2.5.2.1 Types of Tea

Types of tea differ in the plants from which the tea is derived, method of processing and packaging of the tealeaves (Strand Tea Company, 2006; Tea dictionary, 2006; Cheng, 2006; <http://www.2basnob.com>, 2006). Based on the plant from which the different teas are made, they can be broadly classified into regular teas and herbal teas.

- Regular teas

These are the most common types of tea available, also termed “regular tea”. Regular teas are made from the leaves of the plant *camellia sinensis*. Regular teas are further classified into four different types depending on the way in which the leaves are processed; they include (1) White, (2) Green, (3) Oolong and (4) Black teas (Tea dictionary, 2006; Cheng, 2006; Types of tea, 2006).

- Herbal tea

Herbal teas, properly called "herbal tisanes", are brewed from the roots, stems, bark, leaves and flowers of other plants, many of which taste good and or have reported health benefits (Strand Tea Company, 2006). The popularity of herbal health teas has increased significantly due to the presumed additional health benefits of these teas. Examples of herbal teas include rooibos tea, chai teas (Strand Tea Company, 2006; Tea dictionary, 2006 and <http://www.2basnob.com>, 2006).

### 2.5.2.2 Rooibos Tea

Rooibos (*Aspalathus linearis*) is a red herbal tea that is indigenous to the Cape Town region of South Africa, with a sweet and nutty taste. Rooibos is an African slang word of Dutch origin meaning “Red Bush (Afrikaans Rooi-red; bos-bush)”, it is also called a “fine bush” (Marnewick *et al*, 2000; <http://www.africantea.com>, 2006). Rooibos tea is a popular health beverage especially in South Africa and some have referred to it as a great

choice of drink for health-conscious people, as it contains no harmful stimulants and no caffeine with only trace amounts of tannins but is rich in vitamins and minerals (Marnewick *et al*, 2000; Strand Tea Company, 2006; <http://www.africantea.com>, 2006)

Some of the presumed benefits of rooibos tea include

- Does not contain colors, additives or preservatives; making it a natural beverage
- Rooibos tea is naturally caffeine free
- Contains additional antioxidative and reactive oxygen species with a scavenging potency.
- Rooibos tea has been shown to aid in health problems such as insomnia, irritability, headaches, nervous tension, hypertension, relieving stomach cramping, colic in infants, treatment of allergies (such as hay fever, asthma and eczema), certain skin conditions, can help slow the aging process, boosts the immune system, treatment of kidney stones and for several other health benefits.
- Rooibos tea also contains several other minerals such as copper, iron and potassium, calcium, fluoride, zinc, manganese and magnesium (for the nervous system)

(Marnewick *et al*, 2000; Marnewick *et al*, 2005; Strand Tea Company, 2006; <http://www.africantea.com>, 2006 and <http://www.2basnob.com>, 2006).

As rooibos tea is a popular tea especially in South Africa, this study tested its effect on the staining of provisional restorative materials as the tea is derived from a different plant compared to the regular teas.

### **2.5.2.3 Glen Tea ([www.unilever.co.za/ourbrands/foods/glen](http://www.unilever.co.za/ourbrands/foods/glen), 2006)**

Glen tea is a black tea. Like other regular teas, it is made from the leaves of the plant, *camellia sinensis*.

Glen tea is South Africa's largest selling black teabag brand, manufactured by the Unilever Company. It has been delivering its strong and fresh flavour for more than 60 years. Compared to other regular tea brands in South Africa, Glen Tea is of an affordable price and thus a popular brand teabag in South Africa.

Benefits of Glen tea/black tea include

- Black tea contains more anti-oxidants compared to most fruits
- Flavinoids found in Black tea are more effective compared to vitamin C, E and Beta Carotene



## **CHAPTER 3**

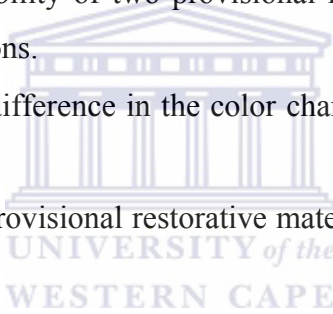
### **AIMS AND OBJECTIVES**

#### **3.1 AIM OF THE STUDY**

The aim of this study was to investigate the color stability of two provisional restorative materials upon exposure to different tea staining solutions and to evaluate the effectiveness of a polishing technique to remove the tea stains from the provisional restorative materials.

#### **3.2 OBJECTIVES OF THE STUDY**

- To determine the color stability of two provisional restorative materials exposed to different tea staining solutions.
- To determine if there is a difference in the color change caused by the different teas tested
- To assess if polishing the provisional restorative materials after tea staining improves on the color change.



#### **3.3 STATEMENT OF THE PROBLEM**

The color stability of tooth-colored restorative materials for provisional restorations is of primary importance when provisional prostheses are used for an extended period of time or when they are used in the aesthetic zone. As such, there is a need to determine the color stability of provisional restorative materials and to determine the effectiveness of a polishing technique to improve the color of the stained provisional restorations.

#### **3.4 HYPOTHESIS**

- There is no significant difference in the color stability of different materials used as provisional restorations for fixed partial dentures when exposed to a staining solution such as tea.

- There is no significant color change when provisional restorative materials are polished after staining by different types of tea.



## CHAPTER 4

### MATERIALS AND METHODS

#### 4.1- SPECIMEN PREPARATION FOR MEASUREMENT OF COLOR

##### 4.1.1 Study Sample and Sample Size

The two provisional restorative materials routinely used in the fabrication of provisional fixed partial dentures and crowns evaluated for color stability in this study were Snap (figure 4.1) and Protemp 3 Garant (figure 4.2). Details of the study sample are listed in table 4.1.

**Table 4:1 Study sample**

Code	Commercial name	Composition	Polymerisation	Shade	Manufacturer	Batch number
SP	Snap	Polyethyl methacrylate	Autopolymerization	62	Parkell, Farmingdale, N.Y. USA	Liquid-40121 Powder-44999
PT	Protemp 3 Garant	Bis-acryl composite resin	Autopolymerization	A3	ESPE, Seefeld, Germany	229266



**Figure 4.1 Snap**  
Manufacturer: Parkell, Farmingdale, N.Y. USA



**Figure 4.2 Protemp 3 Garant**  
**Manufacturer: ESPE, Seefeld, Germany**

The materials used were not of the same baseline shades. Thirty specimens of dimensions 10 mm in diameter and 2.5 mm thick were fabricated of each material. Color changes were measured for each specimen relative to its baseline color. After storage in distilled water for 24 hours, the thirty specimens of each material were randomly divided into three groups of ten specimens each. A group was randomly stored in one of three solutions namely water as the control and rooibos tea and Glen tea as the staining solutions.

#### **4.1.2 Specimen Preparation**

- Materials used

A polyvinylsiloxane mold with an internal diameter of 10 mm and 2.5 mm high was used to fabricate all the specimens. A polyvinylsiloxane mold was chosen as the mold material, as polyvinyl impression materials are often used as matrices to fabricate provisional crowns and fixed partial dentures using the direct or indirect methods.

Plastic strips and a glass slide were used to cover the mold during fabrication of the specimens. After placement of the material into the mold (figure 4.3), plastic strips were placed to cover the material and to achieve a uniform smooth surface (figure 4.4). A glass slab was then placed over the plastic strip and it was gently pressed against the mold to extrude the excess material from the mold (figure 4.5).

- Dispensing/ placement of materials into the mold and polymerization

Care was taken during fabrication of all the specimens to avoid porosities due to air entrapment in the mold. Protemp 3 Garant material was dispensed by a cartridge/gun into the mold and left to autopolymerise for five minutes according to the manufacturer's specifications (figure 4.3). Snap was hand-mixed (figure 4.6) and a spatula was used to place the material into the mold from one side in an attempt to avoid air entrapment in the mold (figure 4.7). The specimens were allowed to autopolymerize according to the manufacturer's instructions.

Upon polymerisation, the specimens were removed from the mold and examined visually for any porosity. The consistency of the testing surface was also observed to assess complete polymerisation. The Snap specimens had a very smooth surface that had a shiny appearance. The Protemp specimens had a smooth surface too but it was not shiny. Two of the Snap specimens had porosity on the non-testing side due to entrapped air. All the specimens were then placed in a controlled environment for 15 minutes until the completion of polymerization. The samples were stored in a controlled dry environment until all the specimens were manufactured.

After all the specimens were fabricated, the non-testing surface of each specimen was coded (marked with a pencil dot) to ensure that color measurements would be taken on the smooth testing side only. The specimens were then rinsed thoroughly with distilled water to remove any debris before immersion in distilled water for 24 hours.

Figures 4.3 - 4.7 illustrate the mold, placement of the provisional restorative materials, placement of strips and glass slide and fabricated specimens

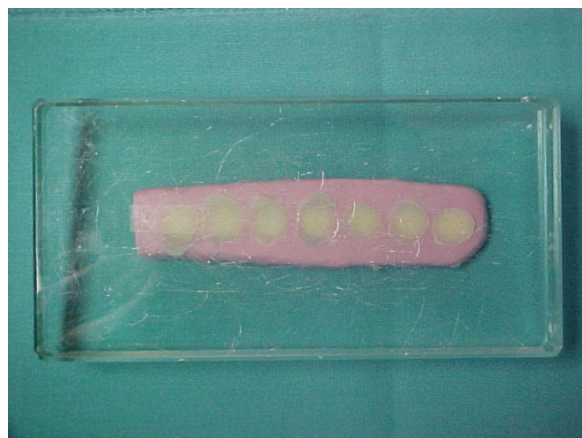




**Figure 4.3: Placement of Protemp 3 Garant in polyvinylsiloxane mold**



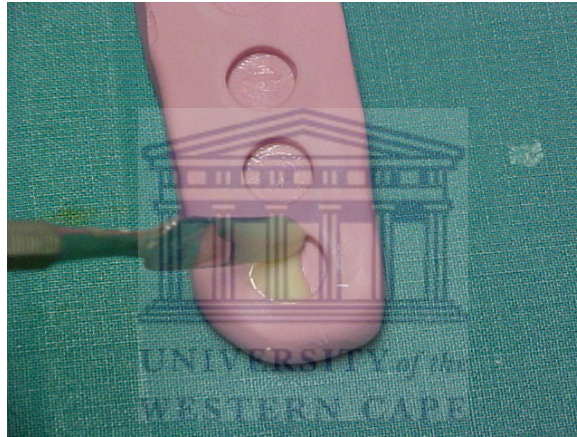
**Figure 4.4: Protemp 3 Garant specimens covered a with a plastic strip**



**Figure 4.5: Placement of glass slab over the plastic strip covering the provisional restorative material specimens**



**Figure 4.6: Mixing Snap**



**Figure 4.7: Placement of Snap into the mold**



**Figure 4.8: Fabricated specimens**

### 4.1.3 Experimental Groups

All 60 specimens were stored in distilled water at room temperature for 24 hours in two plastic containers.

After 24 hours in distilled water, the 30 specimens of each material were randomly divided into three groups of ten specimens each. In all six groups (ten specimens each), the specimens were coded 1 to 10. A group of each material was stored randomly in any of the three solutions namely water as the control solution and the two staining solutions rooibos tea and Glen tea.

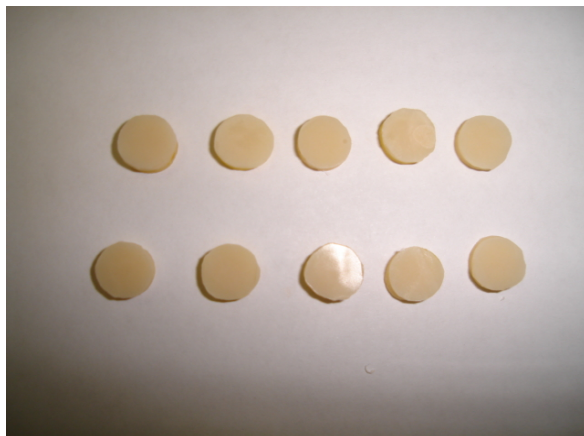
Baseline color measurements were taken for each specimen using a reflection spectrophotometer using the CIE L\* a\* b\* relative to a standard illuminant A against a white background after the 24 hours in distilled water. After obtaining the initial color measurement, each specimen was placed in a correspondingly labeled plastic container containing the respective control or staining agent. A total of sixty coded plastic containers were used to store the sixty specimens representing the six groups.

A flow chart to illustrate the different groups is attached as appendix 1. Table 4.2 lists the six experimental groups. Figure 4.9 shows one of the three groups of the Snap specimens after baseline color measurements and figures 4.10 and 4.11 show the specimen bottles containing the specimens of the three experimental groups for Snap and Protemp.

**Table 4: 2 Experimental groups**

<b>Material</b>	<b>Solution</b>	<b>Experimental codes</b>
Protemp	Rooibos tea	PT-RB
	Glen tea	PT-GN
	Distilled water	PT-DW
Snap	Rooibos tea	SP-RB
	Glen tea	SP-GN
	Distilled water	SP-DW

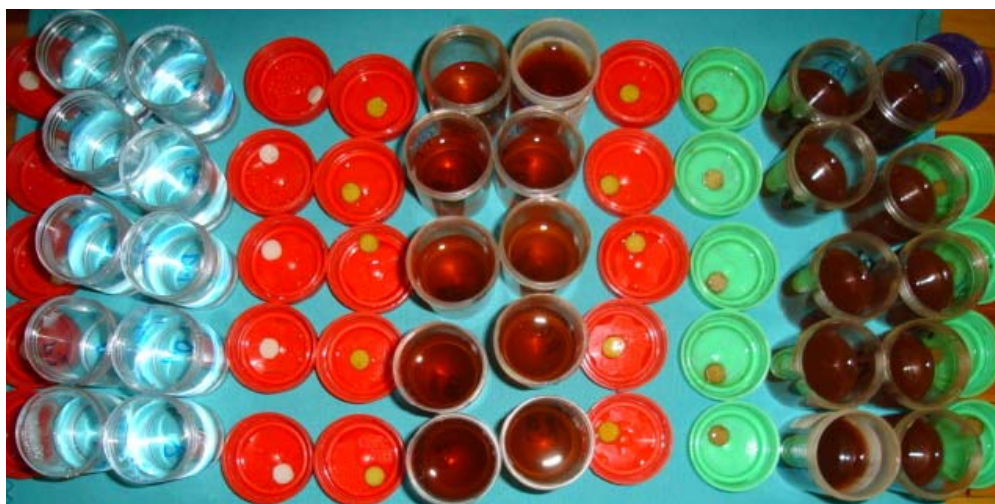




**Figure 4.9: Group 1 Snap specimens after baseline color measurements**



**Figure 4.10: 30 Specimen bottles for the 3 groups of Snap. (SP-RB; SP-GN; SP-DW)**



**Figure 4.11: Protemp groups ready for change of solutions after 1 week (PT-DW; PT-RB; PT-GN)**

## 4.2 STAINING AGENTS

### 4.2.1 Preparation of the Staining Solutions

The staining agents used were rooibos tea (RB) and Glen tea (GN). Distilled water (DW) was used as the control solution. The staining agents used, their concentration and manufacturer's details are listed in table 4.3.

**Table 4:3 Details of staining agents**

Staining solution	Concentration	Brand	Experimental codes
Rooibos tea	10g/l L	Rooibos Tea; Freshpak, South Africa	RB
Glen tea	10 g/1 L	Unilever Bestfoods Robertsons (Pty) Ltd, South Africa	GN

The standard tea solutions (Glen tea solution and rooibos tea solution) were prepared by pouring 5 grams of tealeaves into 500 liters of boiling distilled water (concentration 10 g/L) and allowed to simmer for five minutes. The infusions were filtered through gauze to remove the tealeaves (Sharif *et al*, 2000; Um and Ruyter, 1991). Figures 4.12 to 4.15 illustrate the preparation of the tea solutions.



Figure 4.12: Rooibos tea and Glen tea



Figure 4.13: Boiling tea



Figure 4.14: Filtering tea solution through gauze

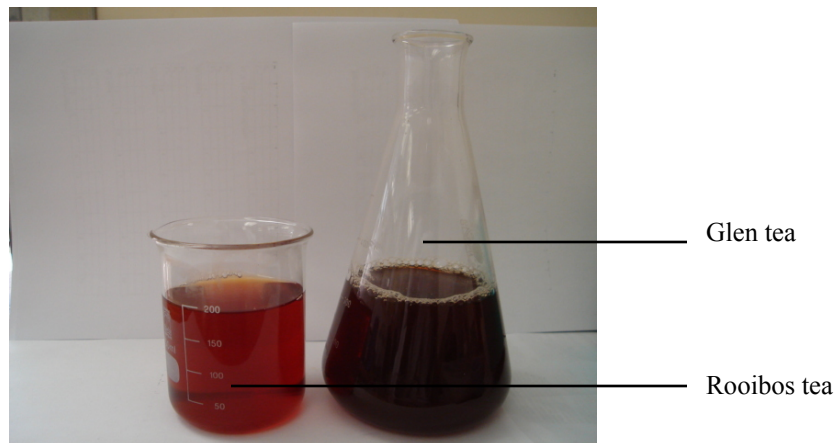


Figure 4.15: Prepared Rooibos and Glen tea solutions (left to right)

The solutions were allowed to cool to room temperature by leaving them to stand for 1 hour after preparation. The solutions were renewed every 2 days.

For the control tests, ten specimens of each material were stored in distilled water for 4 weeks at room temperature. The distilled water was also renewed every two days.

#### **4.2.2 Method of Staining**

Each specimen was immersed individually so that color changes were noted for the same specimen throughout the period of the study. There were two groups of staining agents (Glen tea and rooibos tea) and a control solution (distilled water) for each material (figures 4.10 and 4.11).

Sixty plastic containers divided into six groups (each group consisting of ten plastic containers) were used to store all the specimens during the study. After immersion of the specimens in the respective plastic containers containing the storage solutions, the plastic containers were placed in an orbital shaker moving at a speed of 30 revolutions per minute to prevent air entrapment around the specimens and the settling or sedimentation of the solutions (figure 4.16). The experiments were carried out at room temperature of  $20\pm 3^{\circ}\text{C}$ .



**Figure 4.16: Specimen bottles in the orbital shaker moving at 30 revolutions per minute**



- Cleaning procedure before taking color measurements

Before taking color measurements, all the specimens were removed from the solutions, rinsed with distilled water for 5 minutes and blotted dry with tissue paper. Any accumulated surface sediment was thus removed. Once the specimens had completely dried, color measurements were taken on the testing side of each specimen disc. After color measurements at the specific time intervals indicated, the specimens were re-immersed in the respective storage solutions in their plastic containers.

### **4.3 POLISHING TECHNIQUE AT 4 WEEKS**

After the color measurements were taken at 4 weeks, all specimens previously stored in rooibos tea and Glen tea were polished on the testing side using a coarse pumice and distilled water mixture in a polishing lathe.

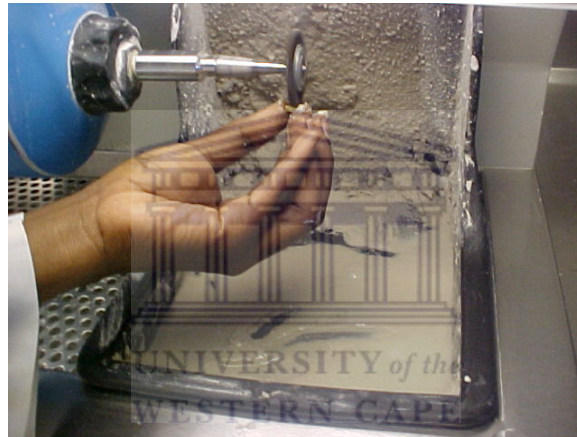
The specimens were polished with pumice and a bristle brush on a dental lathe (KaVo polishing unit EWL 80; KaVo America, Lake Zurich) operating at 1500 revolutions per minute for all polishing procedures for 15 seconds (figures 4.17 and 4.18). Each specimen was polished three times.

The thickness of the polished specimens was determined with a thickness-measuring gauge (Svensen Gauge; Renfert, 1119; 0-10 mm, 0.1 mm, Germany) as depicted in figure 4.19. After the polishing process, the thickness of the specimens was compared to the thickness of the unpolished specimens. The polished specimens were then rinsed in running tap water for 5 minutes and blotted dry with tissue paper. Color measurements were then taken of the polished specimens to assess the efficiency of the polishing process in improving the color of the stained specimens. Photographs (figures 4.20 and 4.21) were taken to illustrate the effect of the polishing process on the stained Prottemp specimens





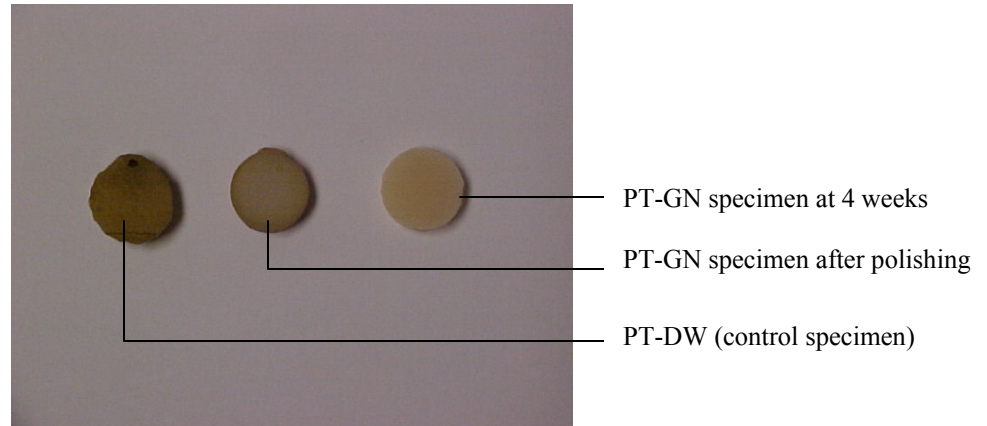
**Figure 4.17 Dental lathe for polishing with a bristle brush**



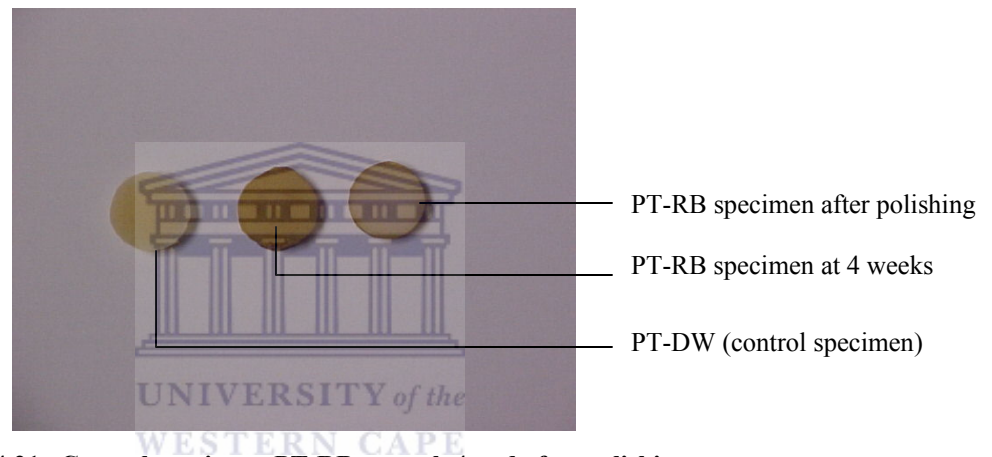
**Figure 4.18 A specimen being polished with a bristle brush**



**Figure 4.19 Renfert gauge measuring thickness of the specimen**



**Figure 4.20 PT-GN at week 4, after polishing and control specimen**



**Figure 4.21. Control specimen, PT-RB at week 4 and after polishing**

## **4.3 COLOR MEASUREMENT**

### **4.3.1 Spectrophotometer Readings**

Color measurements were taken using a reflection spectrophotometer (SP CM-260d Konica Minolta Sensing Inc., Japan-A1005078) using day light (figure 4.23). The testing probe had a measuring aperture of 5 mm in diameter. The aperture of the probe was centered on the specimen to be measured. Before each measurement session, the spectrophotometer was calibrated according to the manufacturer's recommendation by using the supplied white calibration standard A.

The specimens were placed against a white background during the color measurements (figure 4.22). Color measurements ( $L^*$ ,  $a^*$ ,  $b^*$ , coordinates) of each specimen were taken at baseline (after 24 hrs in distilled water), after 7 days, 2 weeks, 3 weeks and 4 weeks in

either the staining solutions or the control solution and after polishing at 4 weeks. Where  $L^*$  represented the values in lightness (black to white),  $a^*$  the values on the red-green coordinate, and  $b^*$  the values on the yellow-blue coordinate. After each session of color measurements, the spectrophotometer was connected to a computer and the data retrieved and stored in an excel spreadsheet.

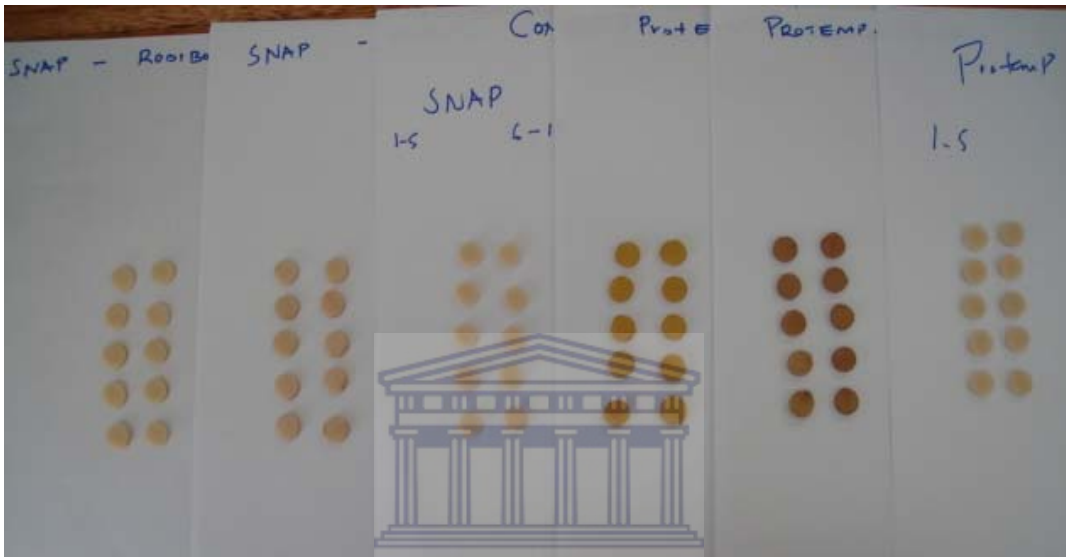
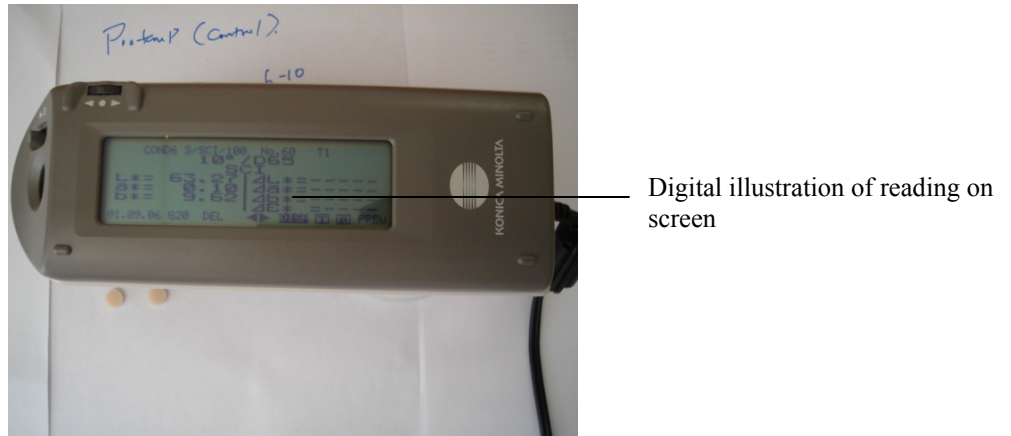


Figure 4.22: Specimens ready for measurements at week 1



Figure 4.23: Spectrophotometer



**Figure 4.24: Taking color measurement of a specimen placed on a white back ground and the reading displayed on the screen of spectrophotometer**

### 4.3.2 Determination of Color and Calculation of Color Changes

The color change was calculated for each specimen at the specific time interval (1, 2, 3, 4 weeks and after polishing) relative to its baseline color. The initial shade differences between the two products were not evaluated and were not related to the significance of the study.

Data for the color measurements were entered into an excel spreadsheet and formulae entered to calculate the color changes for each specimen for all the coordinates ( $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ) at the specific time intervals. For example  $\Delta L_1=L_1-L_0$ ,  $\Delta L_2=L_2-L_0$ ,  $\Delta L_3=L_3-L_0$ ,  $\Delta L_4=L_4-L_0$ , where 1 to 4 corresponds to the values taken at weeks 1 to 4 of the experiment.

The total color change ( $\Delta E^*$ ) was calculated for each specimen relative to its baseline color using the color difference formula:  $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$  where  $\Delta E^*$  represents the color difference and  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  represent the changes in lightness, changes in the red-green coordinate, and changes in the yellow-blue coordinate respectively, after immersion in the various staining solutions or in the control solution.

### 4.4. DATA ANALYSIS

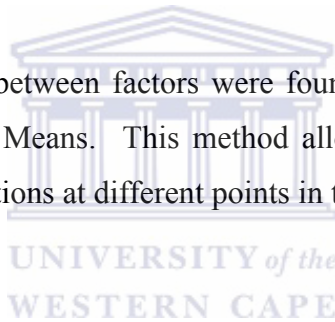
All data was entered into an excel spreadsheet. The color changes in the different CIE  $L^*a^*b^*$  axes ( $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ) and total color changes ( $\Delta E^*$ ) for all the intervals were calculated using an Excel computer software package (Excel 7.0; Microsoft, Redmond, Wash).

The data was analysed using the computer statistical software SAS Version 9 (SAS Institute Inc., Cary, NC, USA). The Mixed procedure was used to perform three-factor analysis of variance (ANOVA), with repeated measures on one factor-time. The 2 between-unit factors were the two materials and three solutions and 1 within-units factor was the six measurement times.

The correlation structure for the repeated factors was modeled using the autoregressive order 1 model. The Mixed procedure allowed to model unequal variances for the different factor levels.

Further analyses were carried out at particular times and for both materials and staining solutions. Two-way ANOVA at each immersion time (WK-1 to WK-4 and after polishing) was used to test for the significant factors involved (materials and solution).

When significant interactions between factors were found, pair-wise comparisons were done using the Least Squares Means. This method allowed for comparisons between materials or between stain solutions at different points in time.



## CHAPTER 5

### RESULTS

#### 5.1. INTRODUCTION

In this study, the color stability of two provisional restorative materials was determined upon exposure to two different tea-staining agents and a control solution. For each material thirty specimens were fabricated and randomly divided into three groups of ten specimens each. Each group was randomly assigned to one of the two staining solutions (rooibos tea or Glen tea) or to the distilled water group as the control. Color measurements were taken for each specimen at baseline and at 1, 2, 3 and 4 weeks and again after polishing at 4 weeks. Color changes in all the axes ( $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$ ) and the total color change ( $\Delta E^*$ ) were calculated for each specimen at the specific time interval relative to its baseline shade.

The mean and standard deviation of  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  and the total color changes for each specific time interval is listed in tables 5.1 to 5.4. The statistical analyses of the results are presented in tables 5.5 to 5.11.

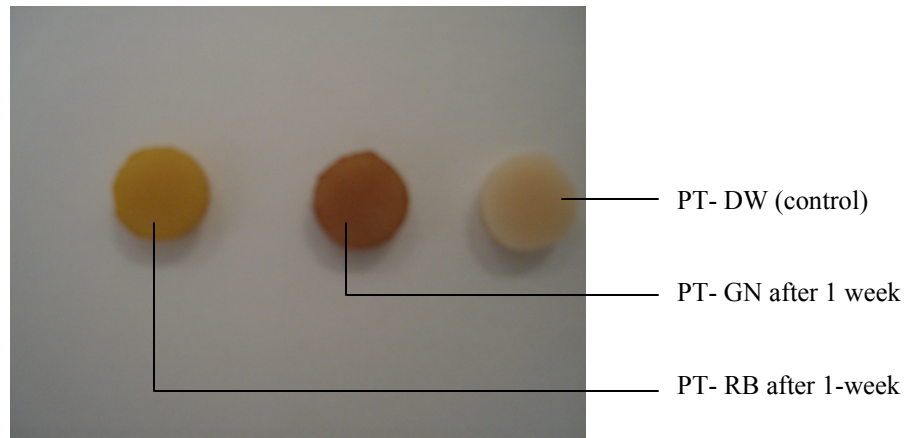
#### 5.2 CHANGES IN THE DIFFERENT COLOR AXES ( $\Delta L^*$ , $\Delta a^*$ and $\Delta b^*$ )

- At one week

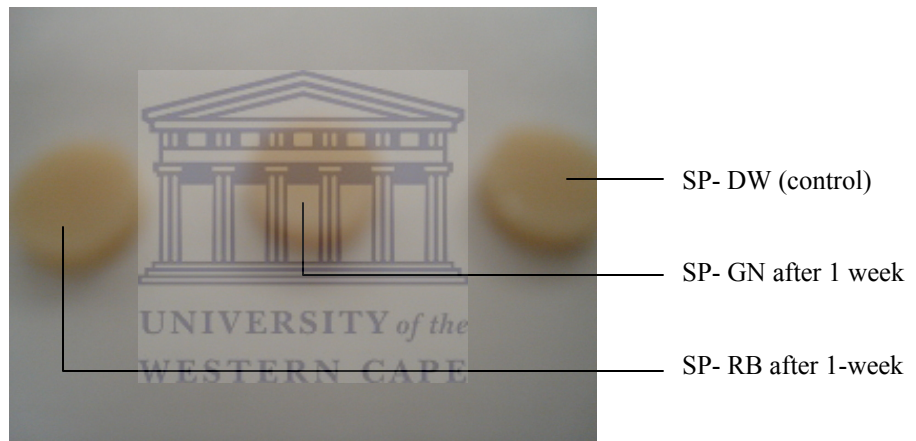
After immersion for one week in rooibos tea the Protemp specimens showed marked changes in the  $b^*$  axis (yellow-blue axis) and the samples had a yellowish discoloration as illustrated in graph 1 attached as appendix 2. While the Protemp samples immersed in Glen tea had changes in both the  $L^*$  and  $b^*$  CIE Lab axes as illustrated in graphs 2 and 3 attached as appendix 2. This color change is also illustrated in table 5.1 and the figures are highlighted. The specimens appeared brownish-yellow as evident in figures 5.1 and 5.2

After one week the Snap specimens in all the solutions had slight changes in all the color axes ( $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$ ) as listed in table 5.2 and the specimens did not show any color changes on visual observation as evident in figure 5.2.





**Figure 5.1 Protomp specimens after immersion for 1 week**



**Figure 5.2 Snap specimens after immersion for 1week**

- Changes in ( $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$ ) across time

The Protomp specimens in both Glen tea and rooibos tea showed small gradual changes in the different color axes after week two, three and four relative to the drastic changes seen after week one (table 5.1). The Snap specimens showed very slight changes in the different axes and no observable color changes were detected visually (table 5.2).

The mean and standard deviations (SD) of the  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  values at the specific time intervals are listed in table 5.1 for Protomp and in table 5.2 for the Snap specimens upon exposure to the two staining solutions and the control solution. These ( $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$ ) values were used in calculating the  $\Delta E^*$  values using the color difference formulae.

**Table 5:1 Means and SD of the  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  values for Protemp experimental groups**

Time	Different color axes	Staining solutions		
		RB	GN	DW
Week 1	$\Delta L1$	-7.5(0.9)	-12.1(1.9)	-0.9(0.5)
	$\Delta a1$	-0.7(0.7)	2.8(1.0)	0.0(0.2)
	$\Delta b1$	22.0(3.3)	8.1(1.5)	-0.4(0.3)
Week 2	$\Delta L2$	-8.7(1.1)	-13.3(2.7)	-0.1(0.5)
	$\Delta a2$	-0.1(0.8)	2.7(1.4)	-0.1(0.2)
	$\Delta b2$	24.8(1.7)	9.9(1.3)	-0.2(0.3)
Week 3	$\Delta L3$	-9.8(1.2)	-13.3(2.5)	0.4(0.4)
	$\Delta a3$	0.7(1.1)	2.4(1.3)	0.0(0.2)
	$\Delta b3$	25.7(1.8)	10.9(1.5)	-0.2(0.2)
Week 4	$\Delta L4$	-11.3(1.4)	-14.0(2.6)	0.1(0.3)
	$\Delta a4$	1.2(1.3)	2.2(1.3)	-0.1(0.2)
	$\Delta b4$	24.9(1.5)	10.1(1.5)	-0.3(0.2)



**Table 5:2 Means and SD of  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  values for Snap experimental groups**

Time	Different color axes	Staining solutions		
		RB	GN	DW
Week 1	$\Delta L1$	0.5(0.8)	1.1(0.5)	0.4(0.5)
	$\Delta a1$	-0.21(0.3)	0.2(0.3)	-0.2(0.2)
	$\Delta b1$	0.2(0.9)	-0.2(0.5)	-0.1(0.4)
Week 2	$\Delta L2$	0.7(0.6)	-0.3(0.8)	0.8(0.4)
	$\Delta a2$	-0.3(0.3)	0.3(0.5)	-0.2(0.2)
	$\Delta b2$	0.7(0.6)	0.1(1.3)	0.0(0.6)
Week 3	$\Delta L3$	1.5(0.6)	0.1(1.3)	1.6(0.5)
	$\Delta a3$	-0.3(0.2)	0.4(0.7)	-0.29(0.3)
	$\Delta b3$	0.5(0.4)	0.1(0.8)	0.2(0.5)
Week 4	$\Delta L4$	0.9(0.8)	-0.9(1.3)	1.0(0.5)
	$\Delta a4$	-0.2(0.2)	0.5(0.8)	-0.1(0.2)
	$\Delta b4$	0.8(0.7)	0.03(1.0)	0.2(0.6)

### 5.3 TOTAL COLOR CHANGES ( $\Delta E^*$ )

#### 5.3.1 Total Color Changes Over Time

There were variations in the total color changes ( $\Delta E^*$ ) for the specimens in the different experimental groups and even slight variations in the total color change ( $\Delta E^*$ ) for specimens within the same experimental group as shown in graphs 4 to 10 attached in appendix 2. The mean color change ( $\Delta E^*$ ) and the standard deviation for the different experimental groups is listed in table 5.3 for Protemp and table 5.4 for Snap over the period of four weeks.

**Table 5:3 Mean and SD of the total color change for Protemp over the four weeks upon immersion in the two staining solutions and the control solution**

<b>Solution</b>	<b>ΔE 1</b>	<b>ΔE 2</b>	<b>ΔE 3</b>	<b>ΔE 4</b>
<b>Rooibos</b>	23.2(3.4)	26.3(1.7)	27.6(1.8)	27.4(1.4)
<b>Glen</b>	14.9(2.5)	16.9(2.7)	17.6(1.7)	17.5(2.2)
<b>Distilled water</b>	1.1(0.4)	0.6(0.3)	0.6(0.2)	0.5(0.2)

**Table 5:4 Means and SD of the total color changes for Snap over the four weeks upon immersion in the two staining solutions and the control solution**

<b>Solution</b>	<b>ΔE 1</b>	<b>ΔE 2</b>	<b>ΔE 3</b>	<b>ΔE 4</b>
<b>Rooibos</b>	1.1(0.7)	1.2(0.6)	1.7(0.5)	1.4(0.8)
<b>Glen</b>	1.2(0.4)	1.0(0.8)	1.4(0.9)	1.5(1.3)
<b>Distilled water</b>	0.7(0.3)	1.0(0.5)	1.7(0.5)	1.2(0.5)

- Week one

After one week of immersion in the staining solutions the Protemp specimens discolored markedly more than the Snap specimens for all the staining solutions as shown in tables 5.3 and 5.4 and then revealed in the pair-wise comparison in table 5.5 for materials and staining agents. The Protemp-rooibos tea combination revealed the highest  $\Delta E^*$  values with a mean  $\Delta E$  of 23.2. The Protemp-Glen tea combination also had a marked total color change with a mean  $\Delta E$  of 14.9. The Snap specimens showed small color changes for both staining solutions. The mean  $\Delta E$  for Snap-rooibos tea was 1.1 and for the Snap-Glen tea combination was 1.2.

- Week two and week three

At the second immersion period at week two and the third immersion period at week three, the PT-RB and PT-GN combinations showed a gradual increase in the total color change relative to week 1 as shown in the box plots in figures 5.3, 5.4 and 5.5, and in table 5.3. The Protemp-rooibos combination at week two had a mean  $\Delta E$  of 26.3 and at week three a mean  $\Delta E$  of 27.6 whereas the Protemp-Glen combination at week two had a

mean  $\Delta E$  of 16.9 and at week three the mean  $\Delta E$  was 17.6. The Snap specimens showed slight changes in color with a mean  $\Delta E$  of 1.2 for rooibos and 1.0 for Glen tea at week 2 and  $\Delta E$  of 1.7 for rooibos and 1.4 for Glen tea at week 3. This is also illustrated in the box plot in figure 5.3, 5.4 and 5.5, and in table 5.4. The Protemp-rooibos combination continued to show the highest values of total color change with a mean  $\Delta E$  of 27.6 at three weeks as seen in table 5.3.

- Week four

At week four, the Protemp specimens revealed the highest  $\Delta E$  values for both staining solutions. The Protemp-rooibos combination had a mean  $\Delta E$  of 27.4 with a standard deviation of 1.4 while the Protemp-Glen combination had a mean  $\Delta E$  of 17.5 with a standard deviation of 2.2. Most Snap specimens at 4 weeks showed imperceptible color changes, with the Snap-rooibos combination having a mean  $\Delta E$  of 1.4 with a standard deviation of 0.8 and the Snap- Glen combination having a mean  $\Delta E$  of 1.5 with a standard deviation of 1.3.

- Specimens in distilled water

For both materials, the control specimens immersed in distilled water showed slight changes in color with a mean  $\Delta E$  not exceeding 1.7 for any of the materials immersed in distilled water. Although at week four the Snap specimens in distilled water had a mean  $\Delta E$  of 1.2 and showed greater color changes compared to the Protemp specimens in distilled water with a mean  $\Delta E$  of 0.5. Pair-wise comparisons in table 5.6 revealed that there was a statistically significant difference in the total color change of both the materials on immersion in distilled water at week three and week four although not perceptible visually.

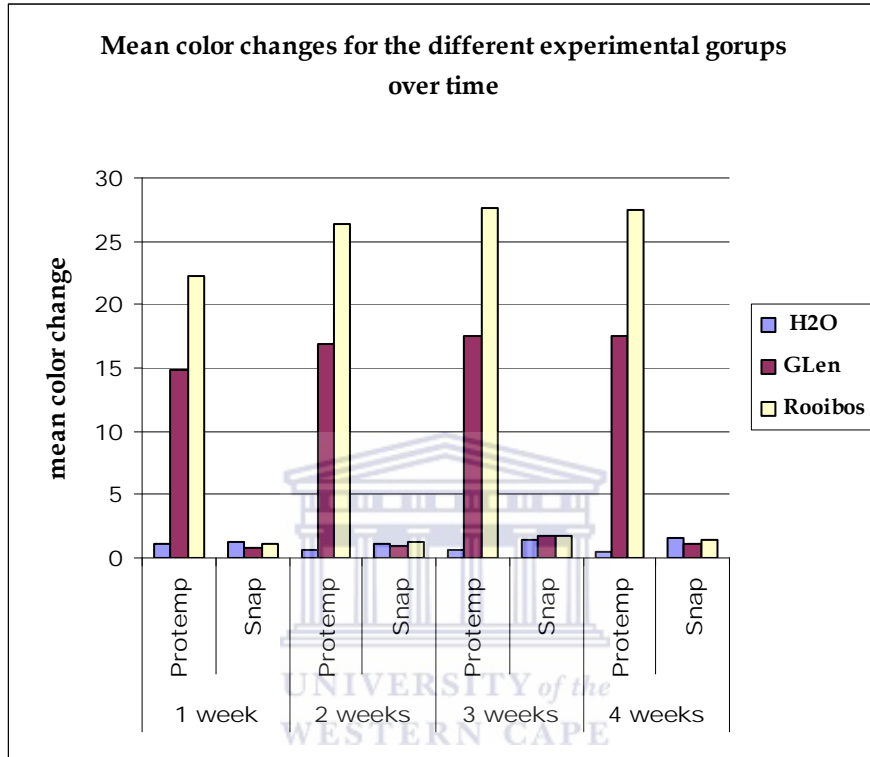
### 5.3.2 Trend of the Total Color Changes

Figure 5.3 graphically shows the mean color changes ( $\Delta E^*$  values) of the experimental groups over the period of 4 weeks for the different staining solutions and the control solution.

Protemp specimens in both Glen tea and in rooibos tea had marked color changes in the first week and showed only slight gradual changes in the following three weeks as

illustrated in the graph in figure 5.3. All the Snap specimens for the different experimental groups showed only slight color changes over the four-week period.

**Figure 5.3 Mean color changes ( $\Delta E^*$ ) of the different experimental groups over time**



### 5.3.3 Box Plot Representation of Total Color Changes

Box plot diagrams graphically illustrate the color changes of the different experimental groups over time. The line dividing each rectangle indicates the median for each experimental group, while the top and the bottom of each rectangle indicates the top 75% and the bottom 25% of the values observed respectively. Thus, the size of the box represents the variance of the total color change in the specific groups with the large boxes indicating wide variance in the total color changes and small boxes indicating small variance in the total color changes for the specific groups. A plus sign (+) inside the box represents the mean value. Abnormally high or low readings are indicated by an asterisk outside the box and are regarded as outliers. Figures 5.4 through to figure 5.6 are box plots illustrating color changes for the different experimental groups over time.

Figure 5.4 Box plot for the total color changes at week 1

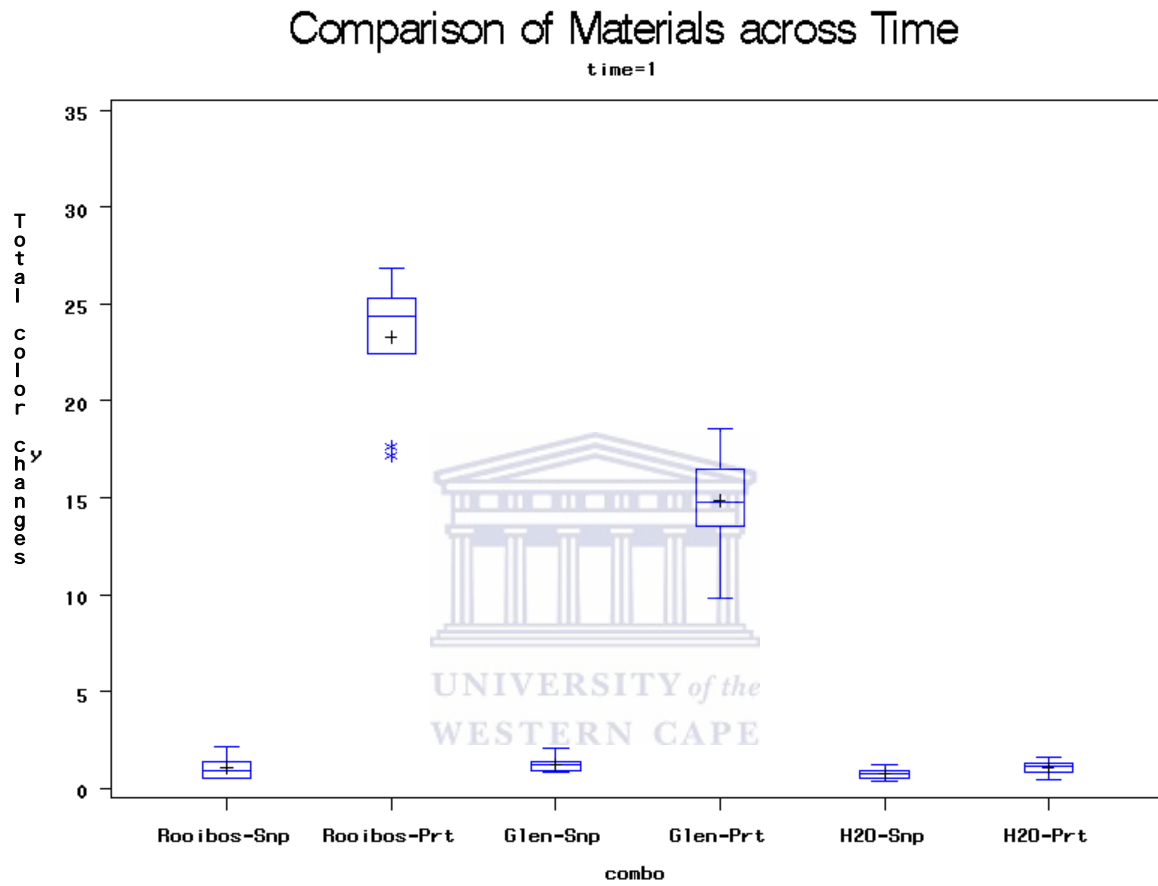


Figure 5.5 Box plot for the total color change at week 2

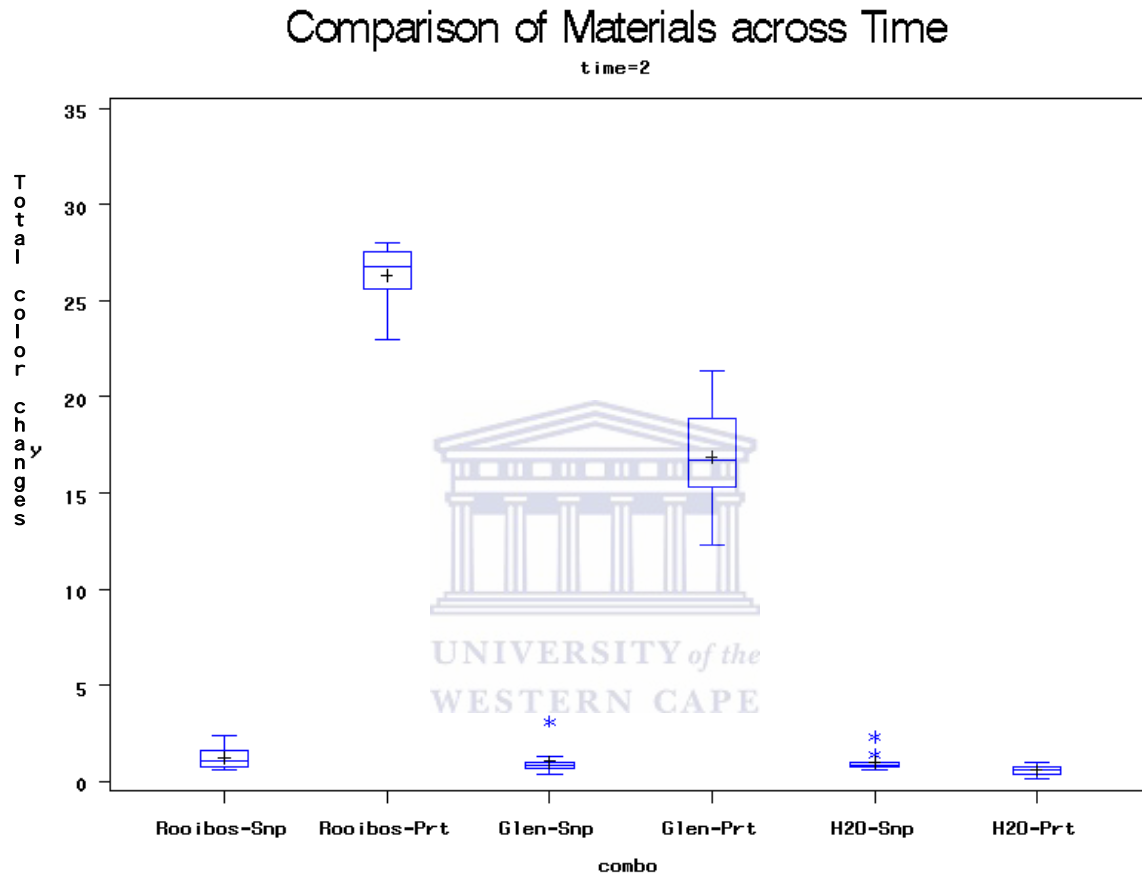


Figure 5.6 Box plot for the total color changes at week 3

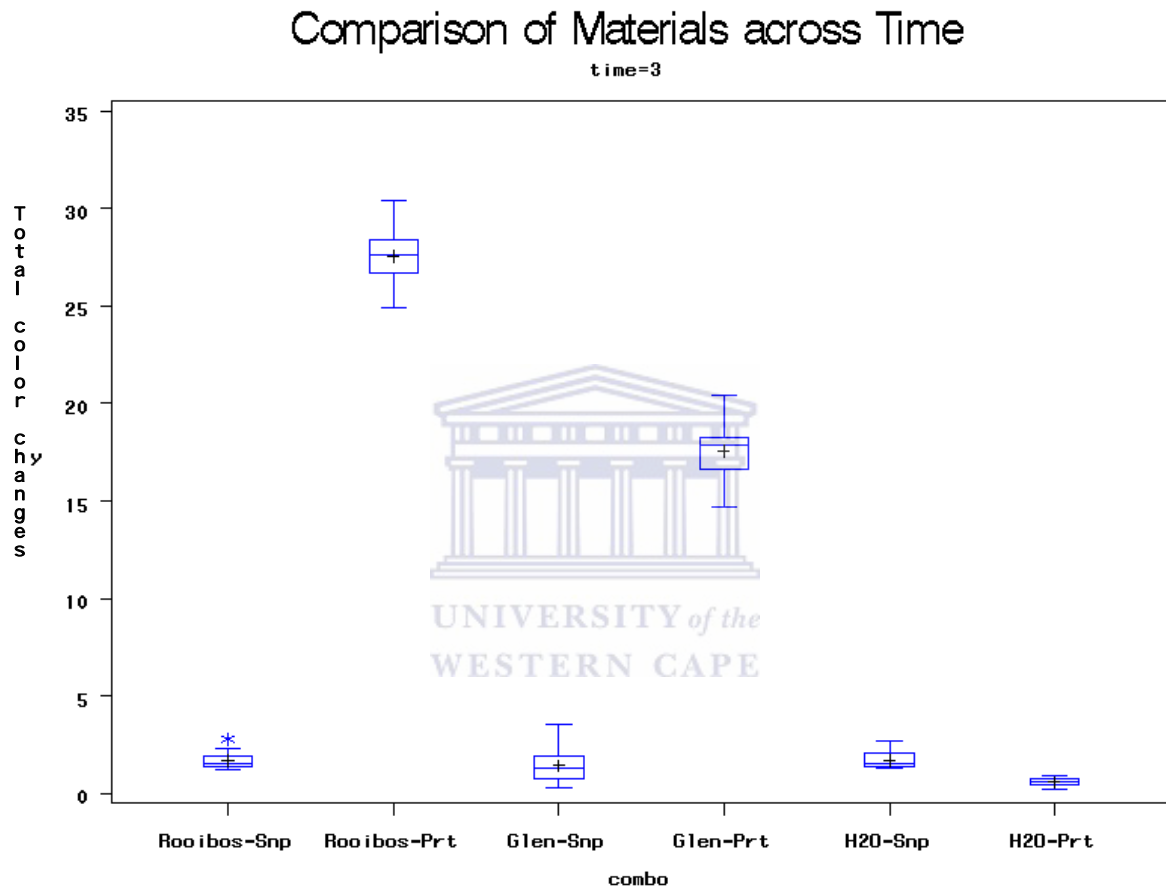
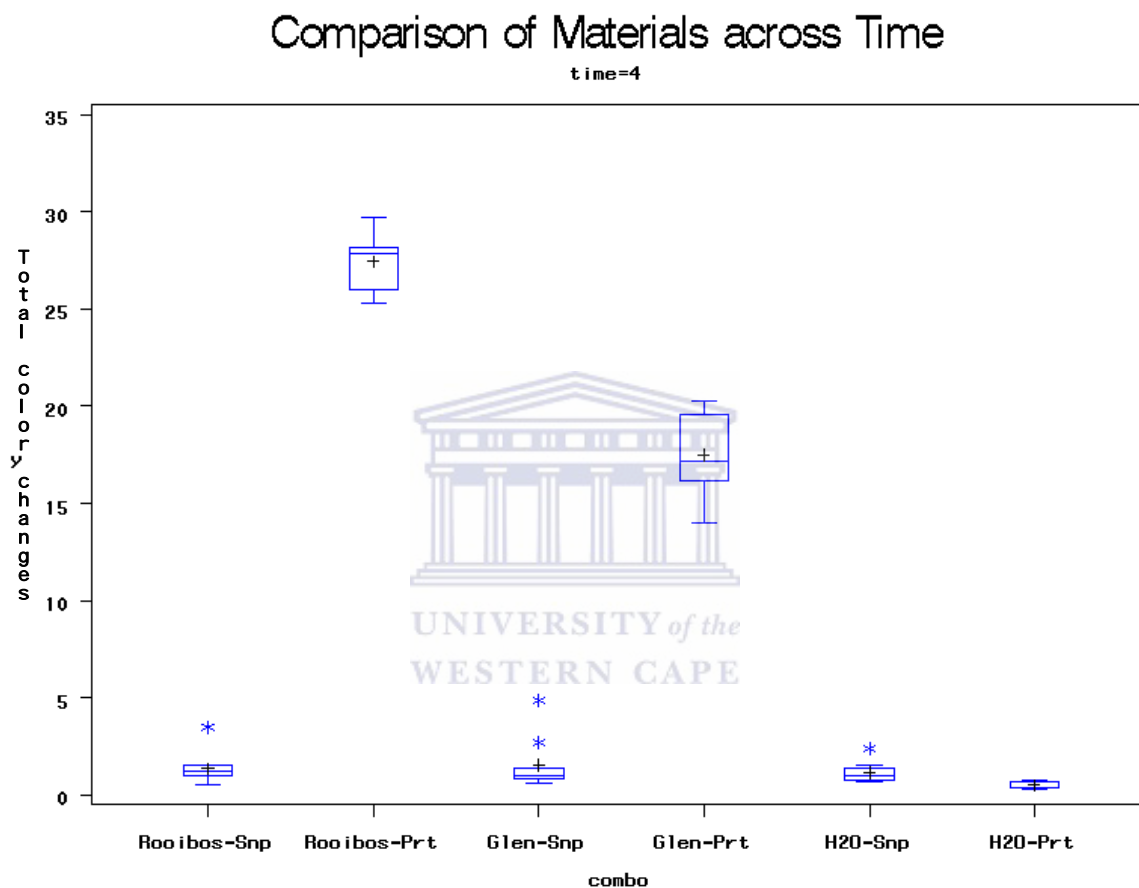


Figure 5.7 Box plot for the total color changes at week 4





There were unequal variances in the total color changes in the different experimental groups as illustrated by the box plots and graphs 4 through to 9 attached as appendix 2. The Protemp specimens immersed in both teas had a wide variance in total color changes within the different groups as illustrated by the wide boxes; while the Snap specimens in all three groups had small variations in the total color changes at all intervals illustrated by the small box plots.

On immersion in distilled water both provisional restorative materials had very small color changes over the four weeks with  $\Delta E^*$  values of less than 3.3. However, at week four as illustrated in figure 5.7 the Snap specimens in distilled water showed slightly more change in color than the Protemp specimens in distilled water as illustrated in figure 5.7. Pair-wise comparison of the two materials immersed in the control solution (Table 5.5- highlighted) revealed a significant difference ( $P < 0.001$ ) in the total color change of the two materials at week three and four.

## **5.4 ANALYSIS OF THE EXPERIMENTAL GROUPS OVER TIME**

### **5.4.1 Repeated measures ANOVA for three factors**

A three-factor analysis of variance (ANOVA) with repeated measures on one factor (time) was carried out to investigate if statistically significant differences ( $P < 0.001$ ) existed between the experimental groups across time. The results are listed in table 5.5 and a summary of the estimated values, the standard deviation, the minimum and maximum values,  $P$  value and  $t$  values are attached as appendix 3.

The results indicate that the effects of all three factors, and all possible interactions among them have differences that are statistically significant ( $P < .001$ ). Since the overall significance of the differences were confirmed, further analyses were carried out at particular times and for particular materials and solutions

**Table 5.5 Type 3 Tests of Fixed Effects (three -way ANOVA)**

Effect	Num	Den	F Value	Pr > F
	DF	DF		
Material	1	54	1964.15	<.0001
Stain	2	54	1043.43	<.0001
Material*stain	2	54	1009.55	<.0001
Time	3	162	16.93	<.0001
Material*time	3	162	8.39	<.0001
Stain*time	6	162	10.55	<.0001
Material*stain*time	6	162	14.02	<.0001

#### 5.4.2 Comparison of Materials

A three-factor analysis (ANOVA) was done to compare the material at the different time intervals (The 2 between-unit factors were the two materials and three solutions and 1 within-units factor was the four measurement times). The results of the analysis revealed that there were statistically significant differences ( $P <.0001$ ) between the two provisional restorative materials for both staining solutions for the duration of the experiment and for distilled water at week three and four as shown in table 5.6 (highlighted). The type of provisional restorative material significantly ( $P <.0001$ ) affected the color stability at each immersion period.

**Table 5:6 Three-way ANOVA for materials (Protemp- Snap)**

Material _material	Stain	Time	Estimate	P Value	Lower	Upper
Protemp Snap	Glen	1	13.63	<.0001	12.11	15.158
Protemp Snap	Glen	2	15.86	<.0001	14.33	17.38
Protemp Snap	Glen	3	16.16	<.0001	14.64	17.69
Protemp Snap	Glen	4	15.96	<.0001	14.44	17.49
Protemp Snap	H2O	3	-1.12	<.0001	-1.44	-0.79
Protemp Snap	H2O	4	-0.68	<.0001	-1.00	-0.36
Protemp Snap	Rooibos	1	22.20	<.0001	20.63	23.77
Protemp Snap	Rooibos	2	25.08	<.0001	23.51	26.65
Protemp Snap	Rooibos	3	25.86	<.0001	24.29	27.43
Protemp Snap	Rooibos	4	26.073	<.0001	24.50	27.64

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### 5.4.3 Comparison of Stains

#### *Protemp*

Pair-wise comparison of the Protemp and the different stain combinations revealed statistically significant differences in the color change of the Protemp specimens exposed to the different staining solutions and the control solution over the duration of the experiment (Table 5.7). Type of staining solution (rooibos or Glen tea) or the control solution significantly ( $P <.0001$ ) affected the total color change of the Protemp specimens at each immersion period.

**Table 5:7 Pair-wise comparison for Protemp in the different staining solutions and in the control solution at different time intervals**

Stain	_Stain	Time	Estimate	Probt	95% confidence interval	
					Lower	Upper
Glen	H2O	1	13.82	<.0000	12.40	15.24
Glen	Rooibos	1	-8.42	<.0001	-10.49	-6.35
Glen	H2O	2	16.31	<.0001	14.88	17.73
Glen	Rooibos	2	-9.42	<.0001	-11.49	-7.35
Glen	H2O	3	16.99	<.0001	15.57	18.41
Glen	Rooibos	3	-9.98	<.0001	-12.05	-7.91
Glen	H2O	4	17.01	<.0001	15.59	18.43
Glen	Rooibos	4	-9.94	<.0001	-12.01	-7.87
H2O	Rooibos	1	-22.23	<.0001	-23.76	-20.71
H2O	Rooibos	2	-25.72	<.0001	-27.25	-24.20
H2O	Rooibos	3	-26.97	<.0001	-28.49	-25.44
H2O	Rooibos	4	-26.94	<.0001	-28.47	-25.42

*Snap*

There were no statistically significant differences ( $P > .02$ ) in the color change of the Snap specimens exposed to the different staining solutions and the control solution over the duration of the experiment (week 1 to 4) as revealed by a pair-wise comparison of Snap and the different staining solutions (table 5.8). Type of staining solution did not significantly ( $P > .02$ ) affect the color stability of the Snap specimens at each immersion period.

**Table 5.8 Pair-wise comparisons for Snap in different staining solution and in the control solution at different time intervals**

Stain	_Stain	Time	Estimate	Probt	95% confidence interval	
					Lower	Upper
Glen	H2O	1	0.51	0.12	-0.13	1.15
Glen	Rooibos	1	0.15	0.68	-0.56	0.86
Glen	H2O	2	0.02	1.06	-0.62	0.66
Glen	Rooibos	2	-0.19	0.60	-0.90	0.52
Glen	H2O	3	-0.29	0.38	-0.93	0.36
Glen	Rooibos	3	-0.28	0.43	-1.00	0.43
Glen	H2O	4	0.37	0.26	-0.27	1.01
Glen	Rooibos	4	0.17	0.63	-0.54	0.89
H2O	Rooibos	1	-0.36	0.15	-0.85	0.13
H2O	Rooibos	2	-0.21	0.41	-0.70	0.29
H2O	Rooibos	3	0.00	0.99	-0.49	0.50
H2O	Rooibos	4	-0.19	0.44	-0.69	0.30

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## 5.5 EFFECT OF POLISHING

### 5.5.1 Thickness of polished specimens

There was no significant change in the thickness of the specimens after the polishing process. As evident from the measurements taken with the micrometer thickness-measuring gauge (table 5.9). Results showed an average change in thickness of 0.2/ 10 mm (0.02 mm) indicating that only the surface layer of the specimens was ground off during the polishing process of the specimens.

**Table 5.9: Thickness measurements in millimeters of the specimens after polishing**

Material-Stain Combination	1	2	3	4	5	6	7	8	9	10
PT-RB	2.5	2.4	2.5	2.5	2.5	2.4	2.5	2.5	2.4	2.4
PT-GN	2.2	2.5	2.5	2.4	2.4	2.3	2.5	2.7	2.5	2.4
SP-RB	2.5	2.5	2.5	2.4	2.5	2.7	2.4	2.6	2.5	2.4
SP-GN	2.5	2.6	2.5	2.4	2.5	2.5	2.6	2.5	2.5	2.5

### **5.5.2. Two factor analysis of the effect of polishing**

The color values of each polished specimen were compared to the color values at week four and at baseline. A two-factor analysis of variance (ANOVA) was carried out to investigate if statistically significant differences ( $P < 0.001$ ) existed after polishing the stained specimens for both materials. The results of the analysis attached as appendix 4 revealed statistically significant differences in the color of the polished specimens as compared to the values prior to the polishing process.

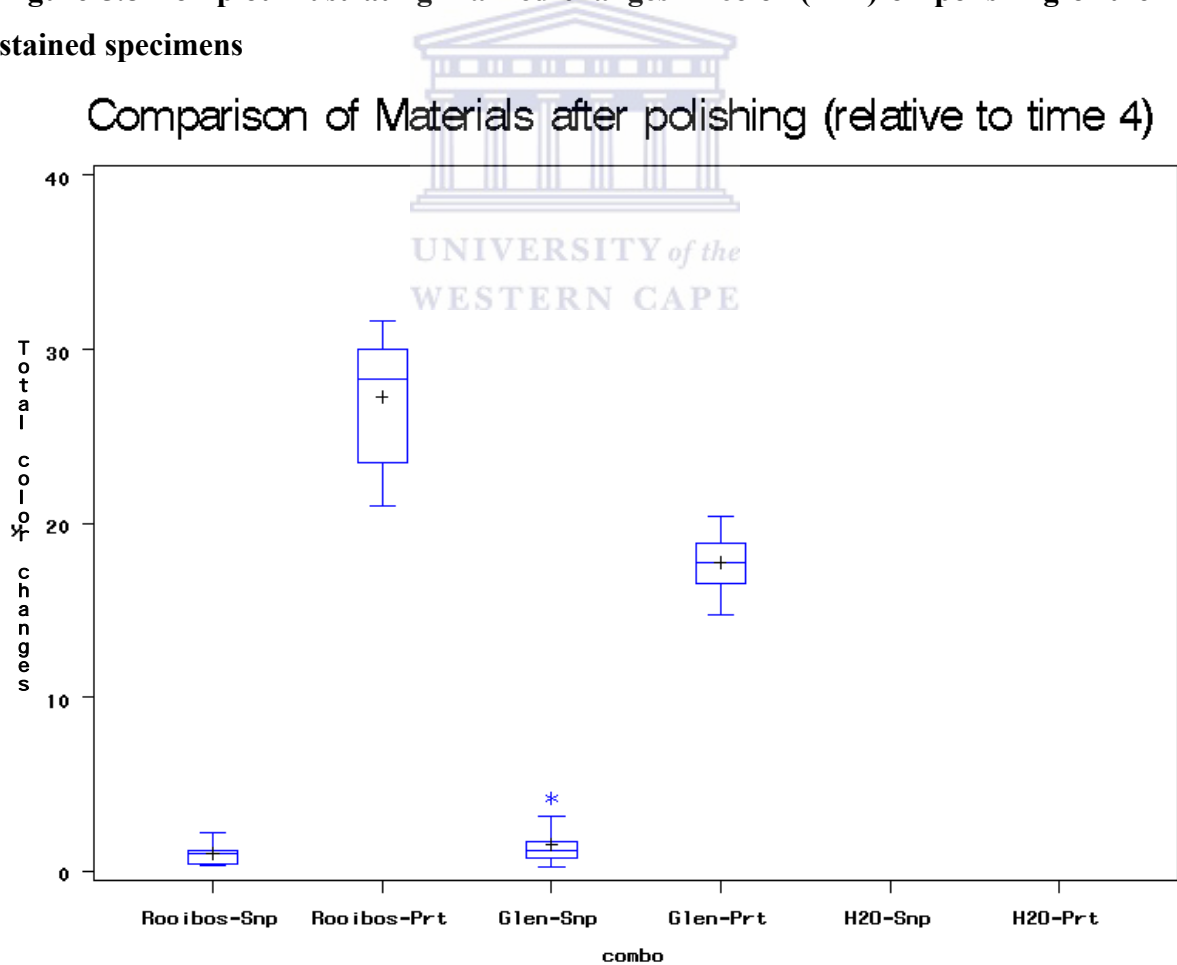
### **5.5.3 Color of Polished Specimens Compared to Week 4**

There were marked color changes after polishing the Protemp specimens that were stained with both the rooibos tea and Glen tea as shown in figure 5.8 with a  $\Delta E$  value of 17.8 for Glen tea and a  $\Delta E$  value of 27.3 for rooibos tea. There were statistically significant differences in the color changes on polishing the stained specimens ( $P < .005$ ) as listed in table 5.10 and attached as appendix 4 (highlighted). Polishing the specimens markedly improved the color of the stained provisional restorative materials without significantly affecting the thickness of the specimens.

**Table 5.10** Estimated mean values of the total color changes after polishing and *P* values compared to week four values

Material stain	Estimate	StdErr	tValue	Probt	95 %confidence level	
					Lower	Upper
Protemp Glen	17.7613	0.5233	33.94	<.0001	16.70	18.82
Protemp Rooibos	27.2625	1.1853	23.00	<.0001	24.86	29.67
Snap Glen	1.5448	0.3905	3.96	0.0003	0.75	2.34
Snap Rooibos	1.0096	0.1851	5.45	<.0001	0.63	1.39

**Figure 5.8** Box plot illustrating marked changes in color ( $\Delta E^*$ ) on polishing of the stained specimens

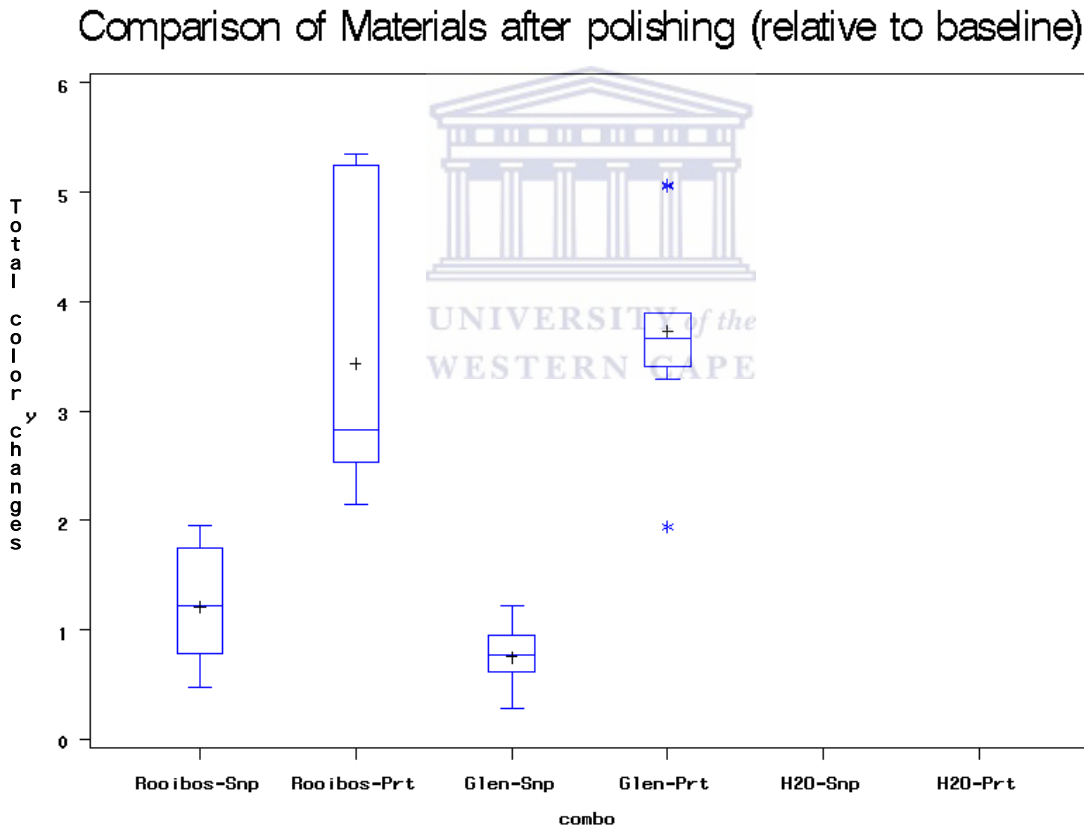


### 5.5.4 Comparison of Polished Specimens to Baseline

On polishing, the discolored specimens had a marked improvement in color. The specimens attained color values near to their baseline shades as shown by the box plot in figure 5.9. After polishing all the specimens had a total color change value of less than five units ( $\Delta E^* < 5.00$ ) compared to the baseline values.

However, there were varying differences in the achieved color, especially for the Protemp-rooibos combination as illustrated by the long box plot. The Protemp-Glen tea combination also showed two outliers as illustrated in figure 5.9.

**Figure 5.9** Box plot illustrating comparison of color of polished specimens to baseline values





### 5. 5.5 Comparison of the effect of polishing on the different tea stains

Table 5. 11 ANOVA for the effect of polishing on the different stains

Material	Stain	_Stain	Estimate	Probt	Lower	Upper
Protemp	Glen	Rooibos	-9.5012	<.0001	-12.13	-6.87
Snap	Glen	Rooibos	0.5352	0.2235	-0.34	1.41

The effect of polishing the stained Protemp specimens was significantly ( $P < .001$ ) affected by the type of staining solution. However, no statistically significant difference was revealed by polishing the Snap specimens ( $P > .2$ ) stained by the two types of tea. The Snap specimens had only slight changes in color at week four.

### 5.6. SUMMARY OF RESULTS

Rooibos tea caused a yellowish discoloration, while Glen tea caused a brownish-yellow discoloration in the Protemp specimens.

Results of the three-factor analysis of variance (ANOVA) with on repeated measures on time indicated that the effects of all three factors (materials, solutions and time), and all possible interactions among them had differences that were statistically significant ( $P < .001$ ).

Pair-wise comparisons revealed that the type of provisional restorative material and solution significantly ( $P < .05$ ) affected the color stability at each immersion period, the interaction of the factors was also significant at each period of time.

Protemp showed marked discoloration for both teas compared to Snap specimens at all time intervals ( $P < .001$ ). The Protemp specimens immersed in both teas showed marked color changes in the first week and thereafter (weeks 2, 3 and 4) the specimens had only slight color changes. At week four, the Protemp specimens had the highest color change for both rooibos tea and Glen tea with a mean  $\Delta E$  value of 27.4 in rooibos tea and a mean  $\Delta E$  value of 17.5. in Glen tea. Snap was more color stable with very slight color changes with the mean  $\Delta E$  values less than 2.0 for all staining solutions. The Protemp-rooibos tea combination showed the highest discoloration with a mean  $\Delta E$  value of 27.4 at week four.

In distilled water, both provisional restorative materials showed very small changes in color with a mean  $\Delta E$  value of less than 1.7. Though at week three and four a two-way analysis of variance for both materials in distilled water revealed statistically significant differences in total color changes ( $P < 0.001$ ) between the two materials.

Polishing of the discolored specimens caused a marked improvement in the color of the stained specimens with a mean  $\Delta E$  value of greater than 15.0. The polished specimens attained color values near to their baseline shades where the total color change was less than five units ( $\Delta E^* < 5.0$ ) compared to the baseline values. However, there were varying ranges of color values attained by the different specimens on polishing. There were no statistically significant differences in the effect of polishing on the stains caused by the two types of tea.



## CHAPTER 6

### DISCUSSION

#### 6.1 INTRODUCTION

Though one of the requirements of provisional restorations is to have good color stability, current provisional crown and bridge materials undergo color changes when exposed to various environmental conditions (Haselton *et al*, 2005). Recent studies by Koumjian *et al*, (1991), Yannikakis *et al*, (1998) and Scotti *et al*, (1997) on color stability of provisional restorative materials have suggested that color stability of these resins is unpredictable over a long period of time with most of the resins showing unacceptable color changes over time. Doray *et al*, (2001) and Haselton *et al*, (2005) suggested that the color changes of the resins are material specific rather than related to a particular category of materials; as such color stability of a provisional material cannot be predicted based solely on the acrylic resin versus resin composite classification available.

This study evaluated the color stability of two provisional crown and bridge restorative materials upon exposure to different tea staining solutions and evaluated the effectiveness of a polishing technique to remove these tea stains.

#### 6.2 INTERPRETATION OF COLOR CHANGES; LEVELS OF PERCEPTIBILITY AND ACCEPTABILITY

The aim of the study was to observe whether the experimental materials showed perceptible color changes upon exposure to staining agents rather than just statistically significant color differences. Perceptible color changes may compromise the clinical acceptability of a provisional restoration (Um and Ruyter, 1991). Various studies (Seghi *et al*, 1990; Ruyter *et al*, 1987; Gross and Moser, 1977; Um and Ruyter, 1991) have reported different thresholds of color-difference values above which the color change is perceptible or unacceptable to the human eye to varying percentages of people.

1. A value of  $\Delta E^*$  of 1 unit is approximately equivalent to a color difference that is just visually perceptible to 50% of observers under controlled conditions, whereas a  $\Delta E^*$  of greater than 2 is detectable to 100% of observers all the time (Seghi *et al*, 1990).

Um and Ruyter, (1991) also suggested that a  $\Delta E^*$  value of 1 unit is ‘visually perceptible’.

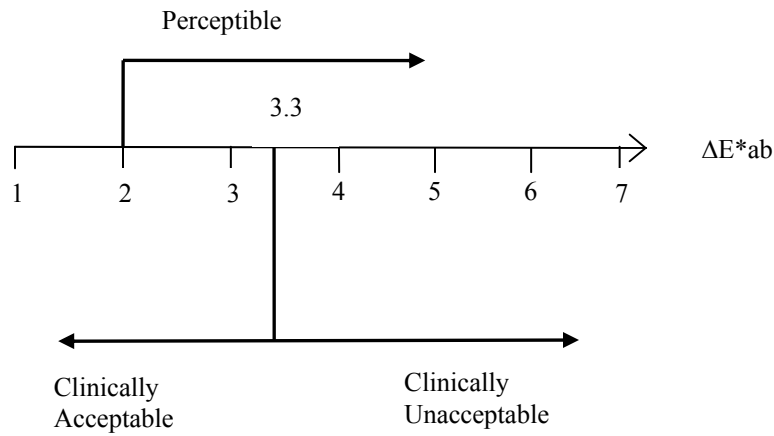
2. However according to Gross and Moser (1977) values of  $\Delta E^*$  between 0 and 2 represent imperceptible color differences, whereas  $\Delta E^*$  values in the range of 2 to 3 represent color differences that are just perceptible visually.

For the purpose of this study a total color change as evident by a  $\Delta E^*$  value greater than 2 ( $\Delta E^* > 2$ ) was stated as visually “perceptible”, whereas a color change ( $\Delta E^*$ ) value of less than or equal to 2 ( $\Delta E^* \leq 2$ ) was considered visually “imperceptible”. Yannikakis *et al*, (1998) used these values in a similar research project.

3. Ruyter *et al*, (1987) and Um and Ruyter, (1991) suggested the upper limit of acceptability in subjective visual evaluation of a perceptible discoloration of a  $\Delta E^*$  value of 3.3. However, Yannikakis *et al*, (1998) and Guler<sup>b</sup> *et al*, (2005) have used a  $\Delta E^*$  value of 3.7 as the upper limit of acceptable color changes based on research done by Johnston and Kao, (1989) and Okubo *et al*, (1998). Johnston and Kao, (1989) evaluated the assessment of an appearance match by visual observation and clinical colorimetry and found that, the average color difference between compared teeth rated as a “match” in the oral environment was a  $\Delta E$  value of 3.7.

In the present study a color change expressed by a  $\Delta E^*$  value greater than or equal to 3.3 ( $\Delta E^* \geq 3.3$ ) was considered visually perceptible as well as clinically unacceptable (Ruyter *et al*, 1987; Um and Ruyter, 1991; Doray *et al*, 1997; Doray *et al*, 2001). Figure 6.1 illustrates selected levels of perceptibility and clinically acceptable limits expressed on a  $\Delta E^*_{ab}$  scale (adopted from Um and Ruyter, (1991) with modification).

**Figure 6.1 Selected levels of perceptibility and clinically acceptable limits expressed on a  $\Delta E^*_{ab}$  scale (adopted from Um and Ruyter, (1991) with modification).**



The results of this study cannot support the null hypothesis as there was a significant difference in the color stability of the two provisional restorative materials tested when exposed to the two tea staining solutions. There was also a significant color change when the provisional restorative material specimens were polished after staining with the different types of tea.

### **6.3 TOTAL COLOR CHANGES**

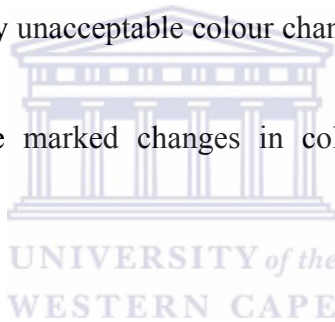
After immersion for one week, there were marked differences in the total color changes displayed by the two materials tested upon exposure to the two teas. The Protemp specimens demonstrated unacceptable colour changes with a mean  $\Delta E_1$  value greater than 14.87 for both staining solutions (Glen tea and rooibos tea) after immersion for 1 week. The Protemp- rooibos combination showed the greatest color changes while most Snap specimens showed imperceptible color changes with a  $\Delta E_1$  value of less than 1.5 for both staining solutions. Two Snap specimens in rooibos tea and one in Glen tea showed just perceptible color changes with  $\Delta E$  values of greater than 2.0 but less than 2.5 as illustrated in graphs 7 and 8 attached as appendix 2.

After immersion in the solutions for 2 and 3 weeks, eighty percent of the Snap specimens still demonstrated imperceptible color changes for both staining solutions. Only two

specimens (one in rooibos and one in Glen tea) showed just clinically unacceptable colour changes as illustrated by graphs 7 and 8 attached in appendix 2. Over the same period the Protemp specimens discoloured even more and showed clinically unacceptable color changes with a mean  $\Delta E$  value of greater than 16.0.

At week four, the Protemp specimens revealed clinically unacceptable color change values for both staining solutions. The Protemp-rooibos tea combination had a mean  $\Delta E$  value of 27.4 with a standard deviation of 1.4 while the Protemp-Glen tea combination had a mean  $\Delta E$  value of 17.5 with a standard deviation of 2.2. Eighty-five percent of the Snap specimens showed imperceptible color changes. The Snap-rooibos combination had a mean  $\Delta E$  value of 1.4 with a standard deviation of 0.8 while the Snap-Glen tea combination had a mean  $\Delta E$  value of 1.5 with a standard deviation of 1.3. Only three Snap specimens (1 in rooibos and two in Glen tea) showed perceptible color changes with two specimens having clinically unacceptable colour changes with  $\Delta E$  values greater than 3.3 but less than 4.0.

Rooibos tea caused the more marked changes in color of the Protemp specimens compared to Glen tea.



#### *Immersion in distilled water*

On immersion in distilled water both provisional restorative materials had imperceptible color changes for the four weeks with values of  $\Delta E^*$  less than 2.2. However, at week four the Snap specimens in distilled water showed slightly more changes in color compared to the Protemp specimens in distilled water and a pair-wise comparison of the two provisional restorative materials in distilled water revealed statistically significant differences ( $P < 0.001$ ) between the materials as regards the total color changes.

#### *Factors affecting color stability*

Results of the three factor analysis of variance (ANOVA) with on repeated measures on time indicated that the effects of all three factors (materials, solutions and time), and all possible interactions among them, were statistically significant ( $P < 0.001$ ).

Pair-wise comparisons revealed that the type of provisional restorative material and solution significantly ( $P < 0.05$ ) affected the color stability at each immersion period, the interaction of the factors was also significant at each period of time.

Findings of this study are in agreement with previous reports by Yannikakis *et al*, 1998 and Guler<sup>b</sup> *et al*, 2005 although it must be noted that different color evaluation techniques, staining agents (exposure conditions) and concentration of staining agents were used. The results from the study indicate that the polyethyl methacrylate Snap is more color stable compared to the Protemp 3 Garant a composite resin based material tested upon immersion in two tea staining agents. With tea and coffee immersion for 30 days, Yannikakis *et al*, (1998) also found the methacrylates (Jet, Caulk TBR, SR-Ivocron PE) to be more color stable compared to materials with different chemistry such as the composite based resin tested-Protemp Garant, Luxatemp Solar, Provipont DC. Crispin and Caputo in 1979 demonstrated that the methacrylates were more color stable compared to other materials tested following immersion in tea-coffee and grape staining solutions for 60 days. Guler<sup>a</sup> *et al*, (2005) found the methacrylate based materials to be more color stable compared to the three composite resins tested for the different polishing procedures following immersion in coffee for 48 hours. In a study to determine the effects of different drinks on stainability of resin composite provisional restorative materials, Guler<sup>b</sup> *et al*, (2005) noted that after 24 hours immersion in tea and coffee solutions, Protemp II specimens like the other resin based composite materials tested showed perceptible color changes.

However, some researchers (Doray *et al*, 2001; Doray *et al*, 1997 and Sham *et al*, 2004) have concluded that composite resins have better color stability. Following *in vitro* accelerated aging conditions, Doray *et al*, (2001) and Doray *et al*, (1997) found some resin-based composites (Integrity, Luxatemp and Protemp Garant) to be as color stable as the acrylic resins tested. Sham *et al*, 2004 also concluded that resin-based composites were more color stable compared to acrylic resins however, they exposed the five provisional restorative materials; three acrylic resins (Trim, Alike, Duralay) and two composite based resins (Luxatemp and Integrity) to different conditions and got differing

results for each experimental condition. On immersion in coffee, the composite based materials revealed more marked color changes compared to the acrylic resins tested while for both water immersion and exposure to ultraviolet radiation the composite based materials were more color stable compared to the acrylic resins tested.

The observations from this study and previous research (Crispin and Caputo, 1979; Doray *et al*, 1997; Yannikakis *et al*, 1998; Doray *et al*, 2001; Guler<sup>a</sup> *et al*, 2005) show that the type of staining agent or exposure conditions are major factors that affect color stability of provisional restorative materials. Results of color stability of provisional restorations after immersion in staining agents like tea or coffee differ from results observed when provisional restorative materials are exposed to accelerated aging conditions or ultra violet radiation. On immersion in staining agents such as tea, coffee, grape juice (Crispin and Caputo, 1979; Yannikakis *et al*, 1998; Guler<sup>a</sup> *et al*, 2005) acrylic resins have better color stability. However, on accelerated aging, exposure to ultraviolet radiation or immersion in water (Doray *et al*, 2001; Doray *et al*, 1997; Sham *et al*, 2004) the composites are more color stable compared to the acrylic resins. The results as regards immersion in water and tea are in agreement with the results of this study.

The rooibos tea caused a yellow discoloration while Glen tea caused a brownish-yellow discoloration. This maybe due to the soluble components of the tea with blue absorption that appears as a yellow colorant, since yellow is the complementary colour to blue (Um and Ruyter, 1991)

Protemp is a chemically cured composite resin-based material. Smales and Gerke, (1992) suggested that the staining of resin-based composite surfaces is a complex phenomenon that can involve several mechanisms.

Tea may stain materials by adsorption of its polar colorants onto the surface of the materials according to Um and Ruyter, (1991). In addition, most bis-acryl polymers are more polar than the methacrylate resin polymers and therefore have a greater affinity towards water and other polar liquids such as tea. This could account for the degree of



color change seen in Protemp 3 Garant when exposed to the different tea staining agents (Haselton *et al*, 2005).

Early discoloration of the provisional restorative materials may also be related to surface smoothness since rough surfaces mechanically retain surface stains more readily compared to smooth surfaces (Guler<sup>a</sup> *et al*, 2005). Though an attempt was made to achieve uniformly smooth surfaces for both materials by allowing the materials to set in contact with matrix strips (Sen *et al*, 2002), the Snap specimens displayed smoother surfaces (very smooth with a shine) compared to the Protemp specimens. This result maybe due to the heterogeneous composition of the composite materials compared to the homogenous composition of the acrylic materials. Presence of filler particles, size distribution of the particles, the composition of the resin matrix and the inherent chemistry of the materials may also influence the surface smoothness (Sen *et al*, 2002 and Guler<sup>a</sup> *et al*, 2005). The relatively rougher surfaces of Protemp, a bis-acryl composite resin compared to Snap an ethyl methacrylate may attempt to explain the marked color changes of the Protemp specimens on exposure to the different tea staining agents compared to the Snap specimens.

Protemp 3 Garant is a chemically cured composite resin-based material thus chemical discoloration of the material itself may also have occurred (intrinsic discoloration). Chemical discoloration is attributed to the oxidation of the polymer matrix or oxidation of the un-reacted double bonds (Yannikakis *et al*, 1998).

#### **6. 4 EFFECT OF POLISHING**

Polishing of the discolored specimens caused a marked improvement in the color of the stained specimens with a  $\Delta E$  value of greater than 15.0. The polished specimens attained color values near to their baseline shades. They had a total color change of less than five units ( $\Delta E^* < 5.00$ ) compared to the baseline values. However, there were varying ranges of color values attained by the different specimens on polishing. There was no statistically significant difference in the effect of polishing for the two types of teas. The results are in agreement with the findings of Um and Ruyter, (1991). However, even after polishing the Protemp specimens still had a significant color change from baseline.

Um and Ruyter, (1991) suggested that the discoloration caused by tea is due to adsorption of colorants from the tea onto the surface of the restorative materials. This can easily be

removed by brushing. Um and Ruyter, (1991) also observed that after 48 hours of immersion in tea, the tea stains were removed by cleansing procedures of soap treatment or combinations of brush and soap treatment or by brush and toothpaste treatment. They also observed that following 1000 hours of immersion in tea, the discoloration was removed by grinding the surface layer of the resin based veneering materials tested that did not have an intrinsic discoloration.

The color changes of the polished specimens compared to their baseline values maybe explained by either absorption of some of the tea stains into the material itself and by intrinsic discoloration of the Protemp material.



## **CHAPTER 7**

### **LIMITATIONS**

The present study had the following limitations.

- The specimen surfaces were flat, whereas, clinically, provisional restorations will have an irregular shape with convex and concave surfaces.
- Furthermore, though very smooth surfaces can be obtained when restorations are allowed to set in contact with matrix strips, clinically it is often necessary to remove excess materials after the fabrication of the provisional restorations with resultant rougher surfaces.
- Tea staining solutions were used in this study to evaluate color stability of the experimental materials; however, provisional materials may be exposed to various other food-staining substances in the oral environment. In addition, other factors could also influence the degree of total color change including thermal cycling and abrasion. These factors should be considered in future studies.

## CHAPTER 8

### CONCLUSIONS AND RECOMMENDATIONS

#### 8.1 CONCLUSIONS

Color stability of two provisional restorative materials was evaluated after 1, 2, 3, and 4 weeks of immersion in different tea staining solutions. The effectiveness of a polishing technique to remove the tea stains was also evaluated in the study. Under the conditions of this study, the following conclusions are made.

- Type of provisional restorative materials, staining solutions and immersion times are significant factors that can affect color stability of the two provisional restorative materials tested.
- Under the conditions of this study, Snap was more color stable compared to Protemp. At 4 weeks, only 10% of the Snap specimens had perceptible color changes.
- Rooibos tea exhibited more discoloration of the Protemp specimens compared to Glen tea.
- Discoloration caused by the teas in this study, could be removed or at least reduced by the polishing of the stained specimens.

#### 8.2 RECOMMENDATIONS

Based on the results of this study, it is recommended that:

- In situations where color stability of provisional restorations is a major factor in the choice of the material, Snap is recommended above Protemp as it proved to be more color stable under the conditions of this study.
- Polishing of discolored provisional restorations may improve the color change.
- Patients be asked to come in regularly for a polishing if the provisional restorations are going to used on a long term basis or are in the aesthetic zone.

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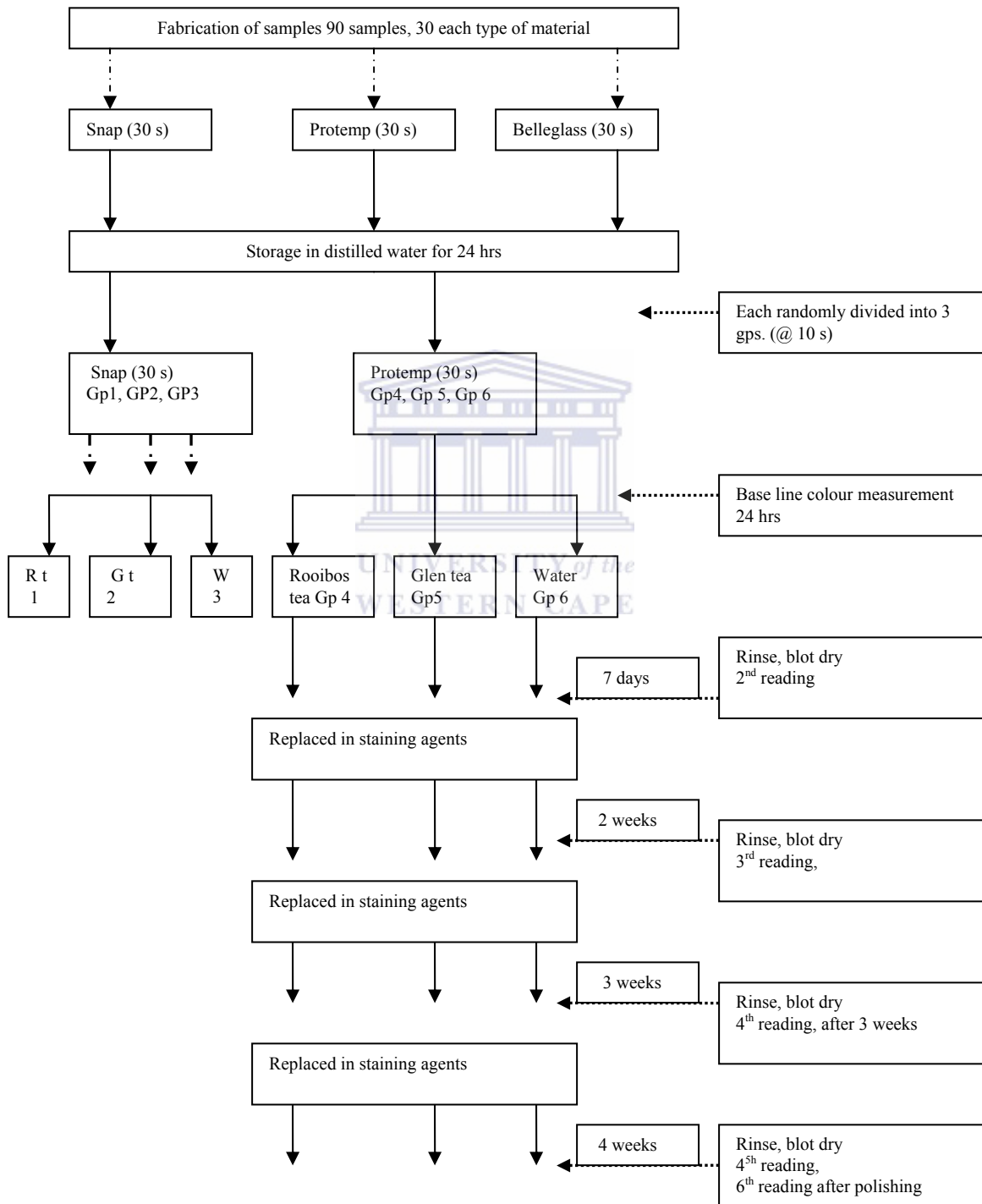
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# 10. APPENDIX

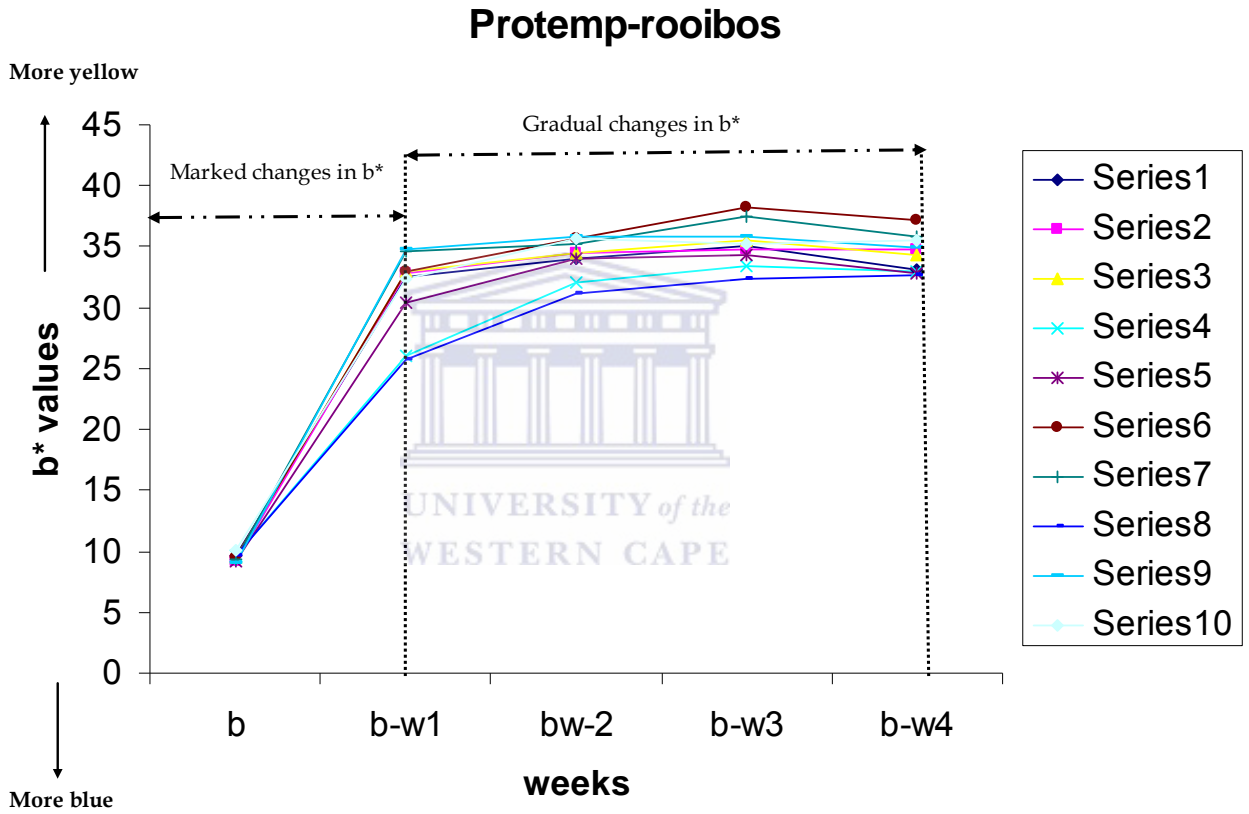
## APPENDIX 1

### Schematic Presentation of Study Design

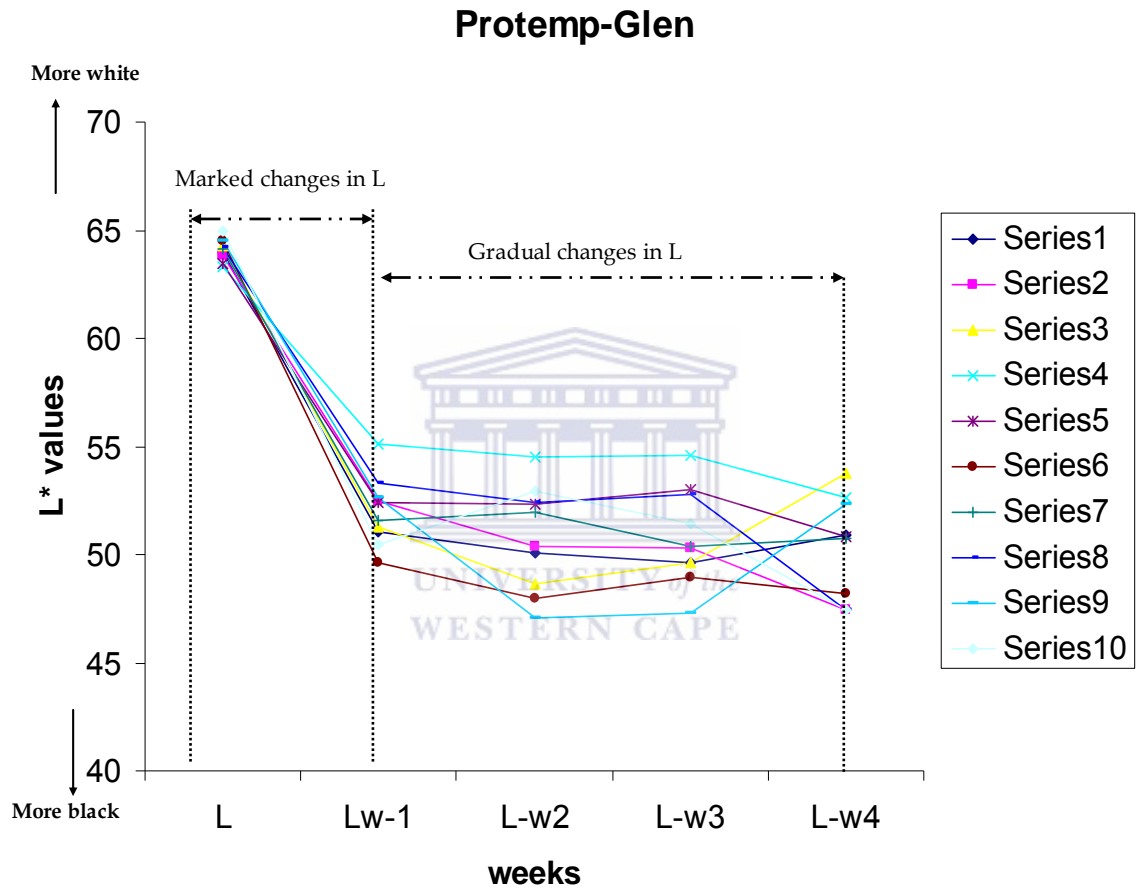


## APPENDIX 2

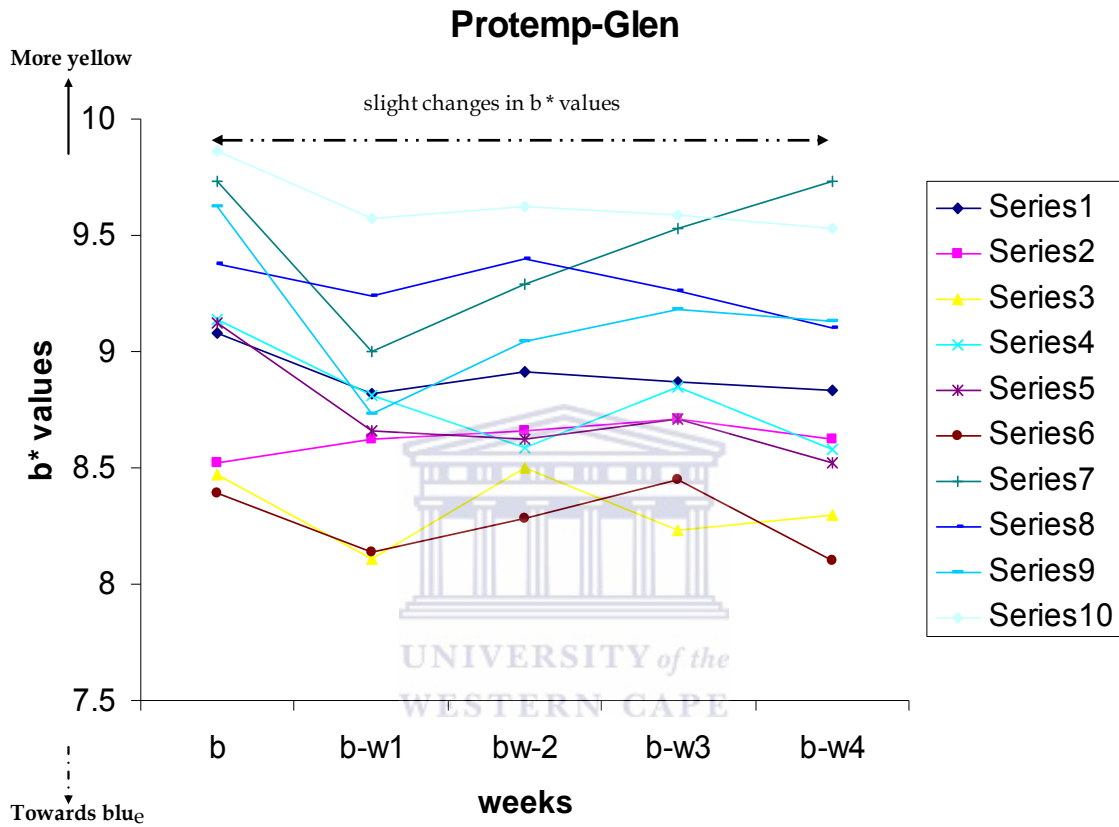
Graph 1. Showing  $b^*$  values of the Protemp-rooibos tea specimens over time



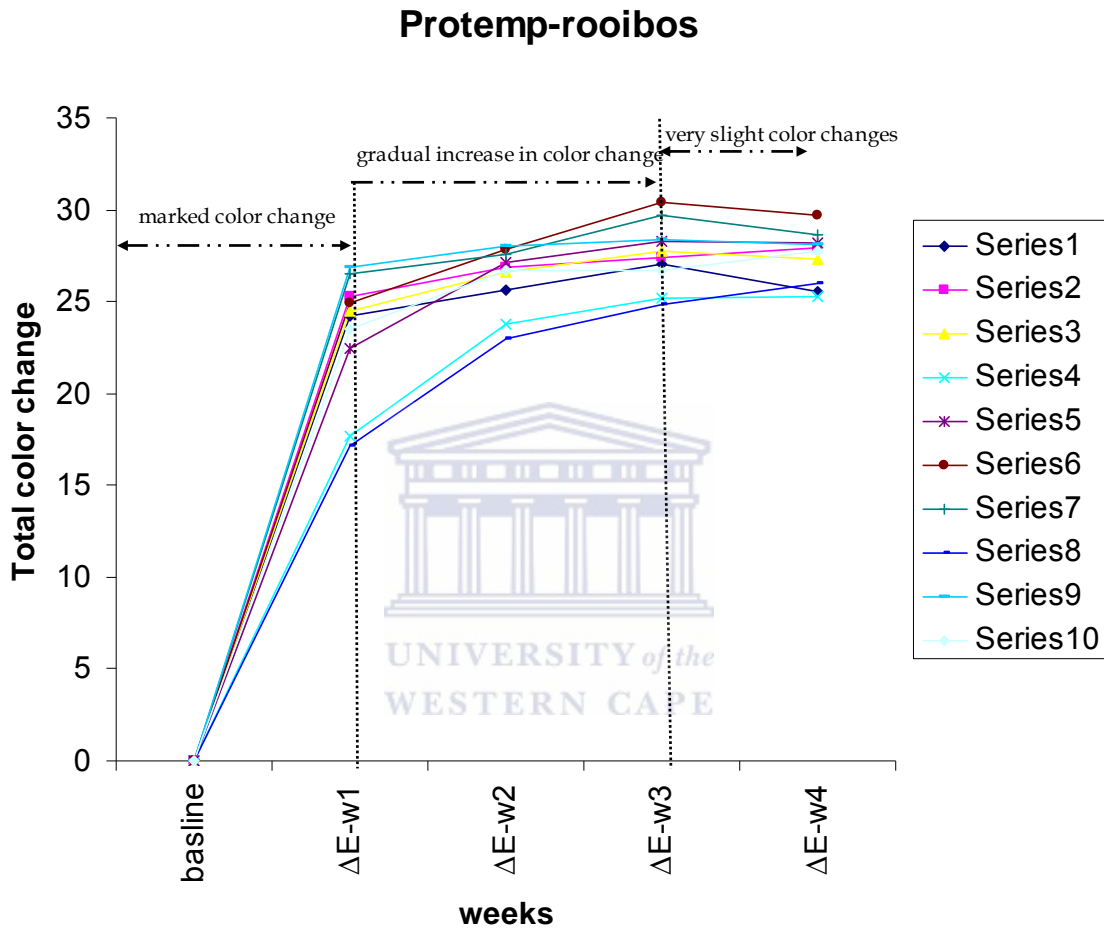
Graph 2. Showing L\* values for the Protemp-Glen tea specimens over time



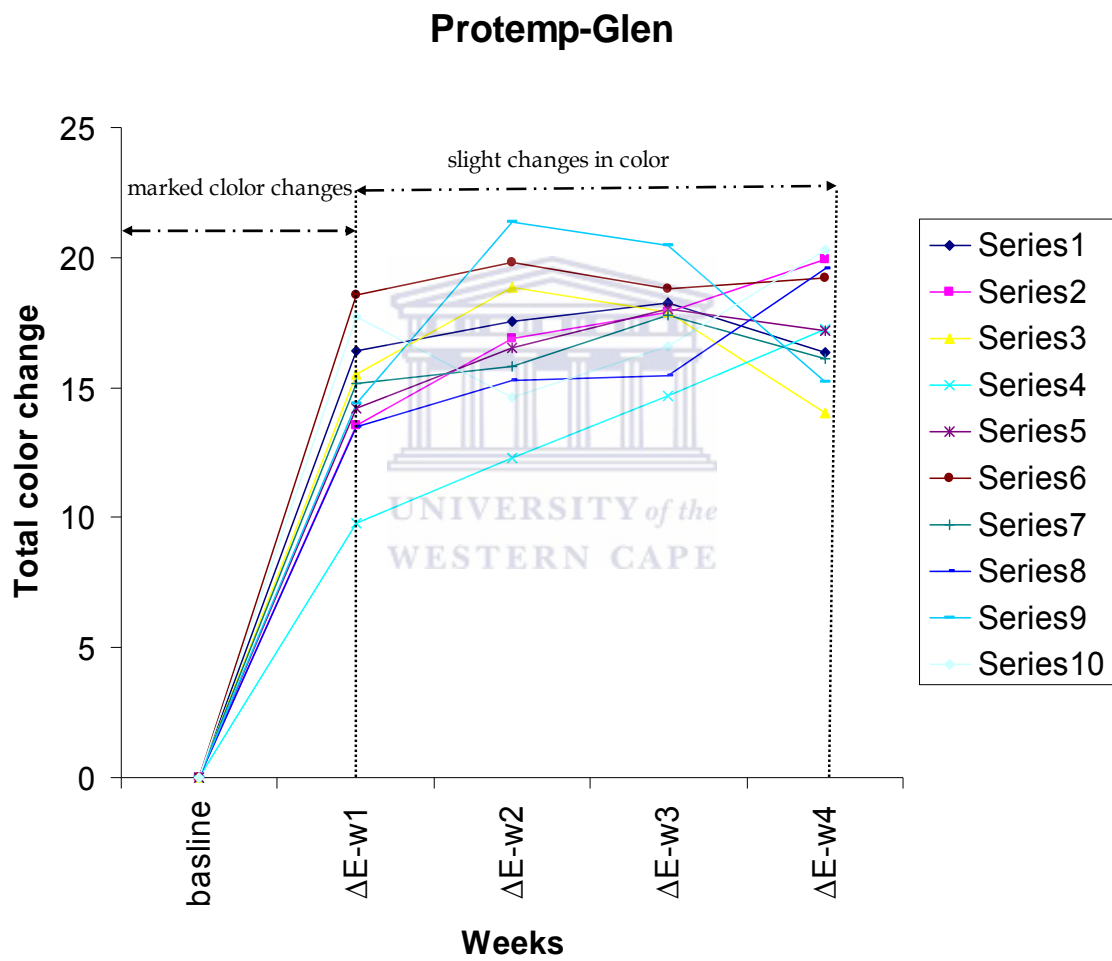
**Graph 3. Showing b\* values for the Protemp-Glen tea specimens over time**



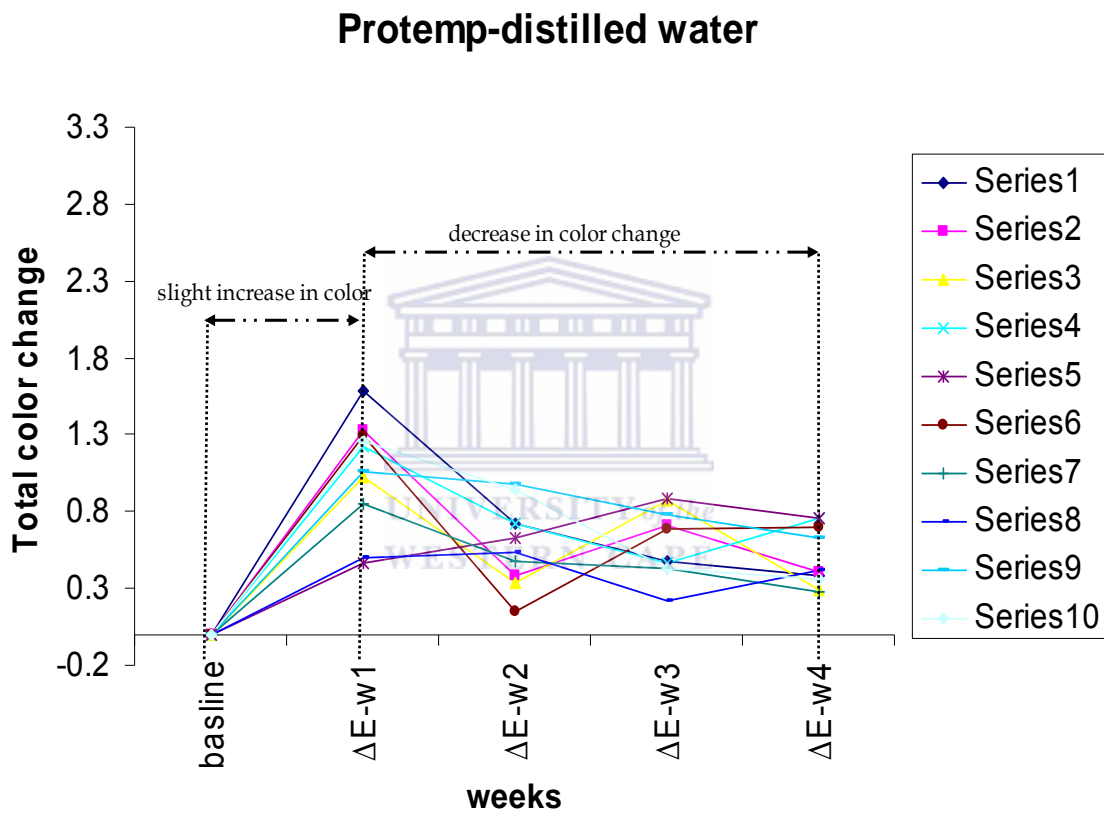
Graph 4. Total color changes for the Protemp-rooibos tea specimens over time



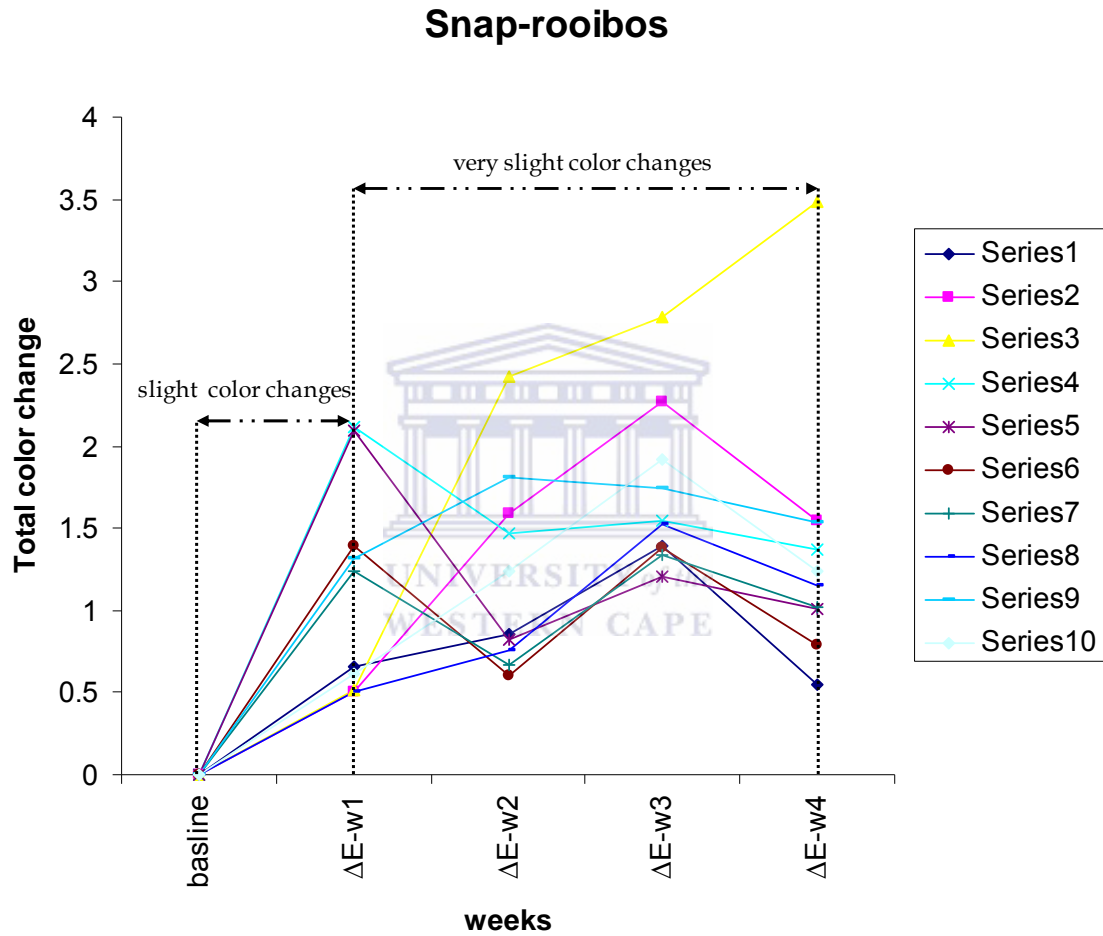
**Graph 5. Total color changes of Protemp- Glen tea specimens over time**



Graph 6. Total color changes for the Protemp- distilled water specimens over time

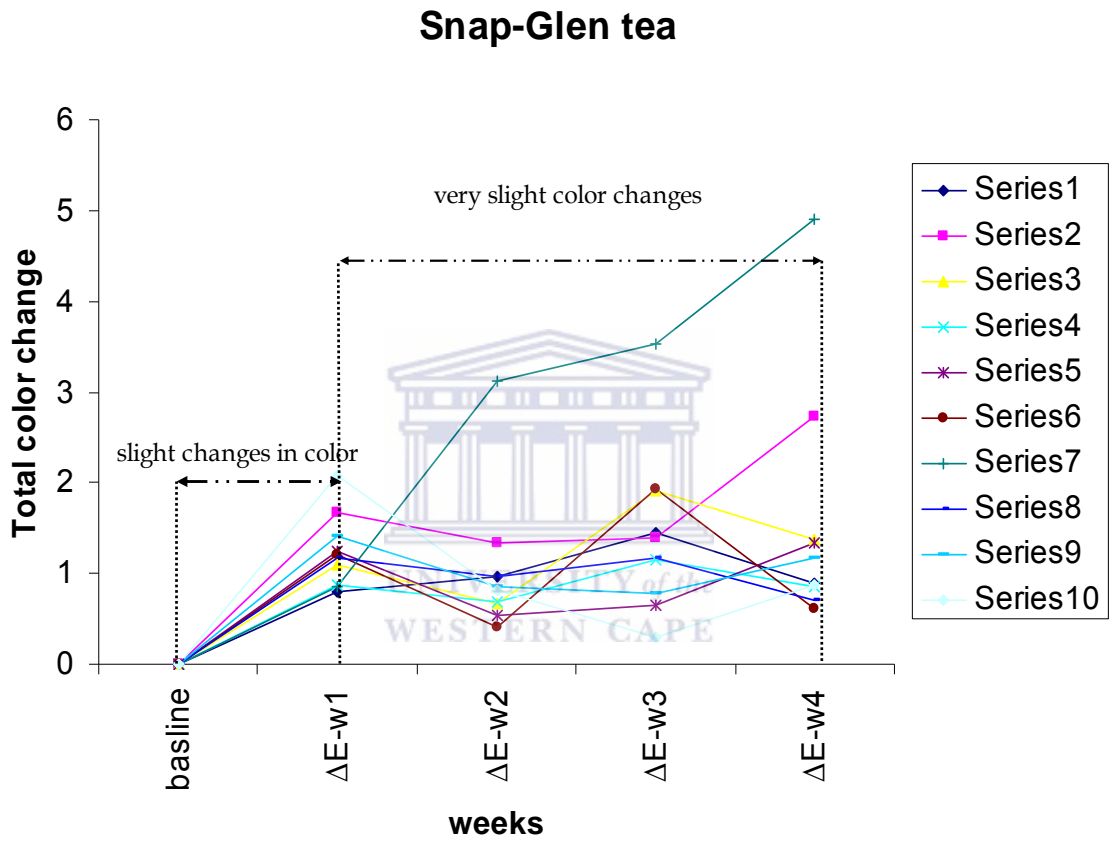


Graph 7. Total color changes for the Snap-rooibos tea specimens over time

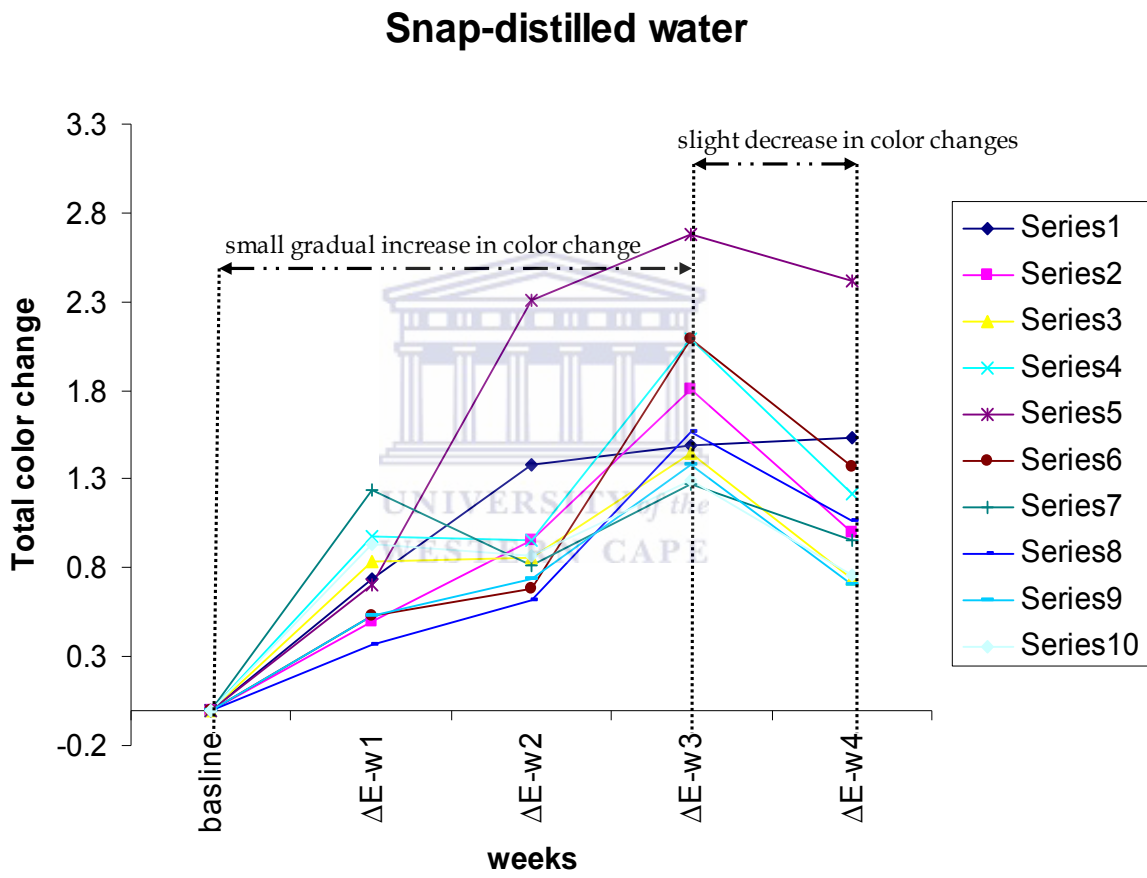




Graph 8. Total color changes for the Snap- Glen tea specimens over time



Graph 9. Total color changes for the Snap-distilled water specimens over time



### APPENDIX 3

#### COMPARISON OF MATERIALS ACROSS TIME

Estimated Mean Values for the Specific Material Stain–Time Combinations.

Material stain	Time	Estimate	StdErr	tValue	Probt	95% Confidence Interval	
						Lower	Upper
Protemp Glen	1	14.88	0.71	20.82	<.0001	13.47	16.29
Protemp Glen	2	16.89	0.71	23.64	<.0001	15.48	18.30
Protemp Glen	3	17.59	0.71	24.61	<.0001	16.18	19.00
Protemp Glen	4	17.50	0.71	24.50	<.0001	16.09	18.92
Protemp H2O	1	1.06	0.08	12.50	<.0001	0.89	1.23
Protemp H2O	2	0.59	0.08	6.92	<.0001	0.42	0.75
Protemp H2O	3	0.60	0.08	7.03	<.0001	0.43	0.76
Protemp H2O	4	0.50	0.08	5.87	<.0001	0.33	0.66
Protemp Rooibos	1	23.29	0.77	30.36	<.0001	21.78	24.81
Protemp Rooibos	2	26.31	0.77	34.29	<.0001	24.79	27.82
Protemp Rooibos	3	27.57	0.77	35.93	<.0001	26.05	29.08
Protemp Rooibos	4	27.44	0.77	35.77	<.0001	25.93	28.96
Snap Glen	1	1.25	0.29	4.24	<.0001	0.67	1.83
Snap Glen	2	1.04	0.29	3.53	<.0001	0.46	1.62
Snap Glen	3	1.43	0.29	4.85	<.0001	0.85	2.01
Snap Glen	4	1.54	0.29	5.25	<.0001	0.96	2.12
Snap H2O	1	0.74	0.14	5.30	<.0001	0.46	1.01
Snap H2O	2	1.02	0.14	7.33	<.0001	0.74	1.29
Snap H2O	3	1.71	0.14	12.32	<.0001	1.44	1.99
Snap H2O	4	1.18	0.14	8.46	<.0001	0.90	1.45
Snap Rooibos	1	1.10	0.21	5.28	<.0001	0.69	1.51
Snap Rooibos	2	1.22	0.21	5.89	<.0001	0.81	1.63
Snap Rooibos	3	1.71	0.21	8.23	<.0001	1.30	2.12
Snap Rooibos	4	1.37	0.21	6.59	<.0001	0.96	1.78

## APPENDIX 4

### EFFECT OF POLISHING

#### COMPARED TO WEEK FOUR

Type 3 Tests of Fixed Effects (three-way ANOVA: material, stain, time)

Effect	Num	Den	F Value	Pr > F
	DF	DF		
Material	1	36	966.83	<.0001
Stain	1	36	43.09	<.0001
Material*stain	1	36	54	<.0001

#### COMPARED TO BASELINE

Effect of polishing compared to baseline (three-way ANOVA)

Effect	Num	Den	F Value	Pr > F
	DF	DF		
Material	1	36	93.92	<.0001
Stain	1	36	0.09	0.7625
Material*stain	1	36	1.93	0.1728

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