THE EFFECT OF DIFFERENT REINFORCEMENTS ON THE

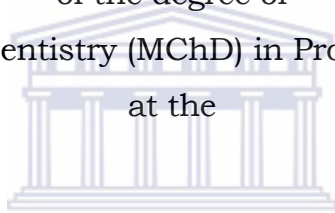# FRACTURE TOUGHNESS OF PROVISIONAL RESTORATIVE

MATERIALS

by

Jan Hendrik Overturf

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of the degree of

Master of Dentistry (MChD) in Prosthodontics

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Supervisor:

Prof GAVM Geerts

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DECLARATION

I, Jan Hendrik Overturf, hereby declare that the work contained in this thesis is my own original work and that I have not previously in its entirety or in part submitted it at any other university for a degree.

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 J H Overturf

Day of _____ of 2006

ABSTRACT

One of the most critical aspects of successful crown and bridgework is temporary restorations. Failure of temporary restorations often affects the patient's confidence and may result in unscheduled appointments for repair. This study compared the fracture toughness of two materials commonly used to fabricate provisional restorations, namely Coldpac®, a polymethyl methacrylate and Protemp 3 Garant®, a bis-acryl composite. It also compared the fracture toughness of the two materials when reinforced with stainless steel wire, glass fibers (everStick C&B fiber®) and polyethylene fibers (Construct® reinforcing braid).

Two Groups (52 samples/group) were prepared from Protemp 3 Garant® (3M/ ESPE, Seefeld, Germany) and Coldpac® (MOTLOID, The Motloid company, Chicago, Illinois, USA) for a three-point bending test conforming to the British Standard 5447. Each group was divided into four subgroups of thirteen specimens each to make provision for the three different types of reinforcement and one control group without any reinforcement. They were stored in distilled water at 37 ^{0}C for 24 hours and tested in three-point bending using a Zwick Universal Testing Machine for peak load to fracture. Specimen deflection was recorded and fracture toughness (K_{IC}) calculated. Medians of the values for the two different resins (controls) and the three reinforcements of each resin were compared (pair wise and otherwise) by means of non-parametric analysis of variance. The

results were compared for statistical significant differences using the Kruskal-Wallis test.

The results of this study showed that:

1. Coldpac® has less fracture toughness than Protemp 3 Garant® (p<0.01).

2. All three reinforcements increased the fracture toughness of Protemp 3 Garant® significantly more than for Coldpac® (glass fiber - p<0.005; polyethylene fiber - p<0.001; steel wire - p<0.005).

3. The glass fibers significantly increased the fracture toughness of both types of provisional materials (p<0.005 for Coldpac® and p<0.0001 for Protemp 3 Garant®). The stainless steel wire also significantly increased the fracture toughness of both provisional materials (p<0.0001 for Coldpac® and p<0.0001 for Protemp 3 Garant®). However, it was found that the polyethylene fibers did **not** increase the fracture toughness of the two provisional materials significantly (p>0.10 for Coldpac® and p>0.10 for Protemp 3 Garant®).

4. For Coldpac® the stainless steel wire increased the fracture toughness significantly more than the reinforcement with polyethylene fibers (p<0.0001), but **not** significantly more than reinforcement with glass fiber (p>0.10). Glass fiber increased the fracture toughness of the Coldpac® significantly more than the polyethylene fibers (p<0.0001). Polyethylene fibers weakened the Coldpac® material, although **not** significantly (p>0.10).

5. For Protemp 3 Garant® the stainless steel wire increased the fracture toughness significantly more than reinforcement with polyethylene fibers ($p < 0.0001$), but again **not** significantly more than glass fiber ($p > 0.10$). Glass fibers increased the fracture toughness of Protemp 3 Garant® significantly more than polyethylene fibers ($p < 0.005$).

DEDICATION

My wife Madeleine, and children Bea, Hugo and Jaco.

ACKNOWLEDGEMENTS

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Chapter 1

Literature Review

1.1 Problem statement

One of the most critical aspects of successful crown and bridgework is provisional restorations. It is the importance of this stage, perhaps more than any other, which is often underestimated. Failure of provisional restorations may affect the patient's confidence and also result in unscheduled appointments for repair. Furthermore, if teeth move during this critical period due to failed provisional restorations, the final restoration may also need to be adjusted or remade.

1.2 Introduction

Auto-polymerising polymethyl methacrylate resins, and more recently bis-acryl composite materials, are most commonly used for indirectly or directly made provisional restorations (Hamza *et al.* 2004). Although the use of pressure during curing increases the fractural strength of polymethyl methacrylate resins, fractures frequently occur in long span provisional restorations (Solnit 1991). Compared with polymethyl methacrylate materials, composite based provisional materials are reported to have a higher flexural strength (>60 MPa) and flexural modulus (>1800 Mpa), however, they also tend to break under clinical loading situations (Lang *et al.* 2003).

Improved physical properties of the provisional materials are required for long span fixed partial dentures (FPDs), for high stress areas, for long-term

provisional restorations or for patients with parafunctional habits. In the past, these properties have been improved by reinforcing the material with wire, wire mesh or fiber mesh. Although several methods to improve bonding of the metal to the resin, such as sandblasting, silanization and metal adhesives were proposed, none of these proved sufficient (Vallittu 1993). Even though this study used denture base acrylic, it is also a polymethyl methacrylate resin. It was then necessary to use the resin material in bulk in order to cover the strengthening material, and failure commonly occurred at the resin/strengthener interface, providing only limited success (Chung *et al.* 1998). Fiber reinforcement has helped to overcome some of these problems, largely by creating a chemical bond between the strengthening fiber and the resin, thus preventing crack propagation (Vallittu 1998). Although carbon and Kevlar fibers were initially used, these have largely been superseded with fibers made of high-density polyethylene, glass or polypropylene in bundles of 10-20 microns, because their appearance is superior (Aydin *et al.* 2002; Hamza *et al.* 2004). The bundles are usually white. However, when wet with the resin they become transparent within the restoration. They are available either as loose, twisted or woven fibers. Woven fibers have a thickness of 0.25-0.50mm and, owing to their multidirectional reinforcement of the resin, provide better strengthening characteristics (Vallittu 1999). According to Kim and Watts (2004) the strength of the resulting reinforced structure is dependent on the volume of the fibers embedded in the resin matrix and the degree of adhesion between the fibers and the polymer. The higher the number of fibers and the better the adhesion, the better the strength characteristics of the material. (Kim and Watts 2004)

In a SEM study done by Kolbeck *et al* (2002) they found that the median of the reinforcing effect of glass fibers was higher than that of polyethylene fibers although not significantly. They attributed this to the difficulty of obtaining good adhesion between the polyethylene fibers and the resin matrix. However, two years later Hamza *et al* (2004) reported no significant difference in the reinforcing effect between polyethylene and glass fibers. This improved performance may have been due to pre-treatment of the polyethylene fibers with silane and plasma (Hamza *et al.* 2004).

1.3 Provisional restorative materials

A provisional restoration should provide both pulpal and periodontal protection, have good aesthetics, sufficient durability to withstand forces of mastication and have good marginal integrity (Hamza *et al.* 2004). A material is chosen for its structural and aesthetic properties, biocompatibility and ease of use. The most commonly used groups of materials to fabricate provisional restorations are the polymethyl methacrylate resins and the bis-acryl composites (Young *et al.* 2001).

1.3.1 Auto-polymerising polymethyl methacrylate resin

This material has been used for many years for provisional crowns and FPDs. Its physical properties include relatively high strength and excellent aesthetics and polish ability (Chung *et al.* 1998). However, according to Gough (1995) it has the following major drawbacks:

1. The polymerisation reaction is highly exothermic. The vital tooth pulp is vulnerable to temperature increases of as little as 5°C and direct fabrication of provisional restorations may cause irreversible damage to it.

2. There is some concern regarding the monomer's direct chemical effect on the pulp.

3. High polymerisation shrinkage causing marginal discrepancies

Lang *et al.* (2003) also reported low mechanical fracture behaviour of these materials. Problems are associated primarily when the direct method of fabrication is used. The indirect method has been associated with superior fit and pulp protection, but it is time-consuming and dependent on the availability of a laboratory (Monday and Blais 1985).

Haselton *et al* (2002) reported that these resins are low-molecular weight, linear molecules that exhibit decreased strength and rigidity and also is not capable of cross-linking with other monomer chains as the bis-acryl resin composites do. Without polymerisation under pressure, air entrapment may occur and result in lower strength values (Solnit 1991).

1.3.2 Bis-acryl resin composite

Gough (1995) reported that bis-acryl resin composites are based on multiple functional methacrylic acid esters similar to the resins used in composites. He also stated that it has a low exothermic polymerisation reaction, a low pulpal irritancy and that the contraction during polymerisation is less than for polymethyl methacrylates. However, this material is less easily added to than methacrylate resins when correcting any marginal defect (Gough 1995).

According to the manufacturer, Protemp Garant® (3M/ ESPE, Seefeld, Germany) was one of the first bis-acryl resin composites used. It has

recently been modified and is now marketed as Protemp 3 Garant®. The modifications include a newly developed monomer system, not with the rigid intermediate chain characteristic of some bis-acryl homologues, but with a somewhat flexible chain in comparison to other synthetic resins (3M ESPE Technical Manual 2004).

This attribute allows a balance between high mechanical strength and limited elasticity of the composite material. According to the manufacturer, the result is a material that can withstand high stresses until fracture and tolerate brief deformation. A study done by Haselton *et al* (2002) confirmed a significant increase in the flexural strength of Protemp 3 Garant® compared to its predecessor. The same study stated that in contrast to polymethyl methacrylate materials, the mixing procedure of composites occurs within the cartridge and may result in more constant material properties. The base pastes of bis-acrylic composite materials consist of bifunctional methacrylates to provide cross-linking for increased mechanical strength and also the application of a cross-linked, highly viscous, bulky monomer improves mechanical properties in restorative and veneering composites (Haselton *et al*. 2002).

1.3.3 Glass fibers

Silanized glass fibers are promising new materials because of their good adhesion to the polymer matrix, high aesthetic quality, and increased strength of the resulting composite (Hamza *et al.* 2004). Researchers have found that the position, quantity, and direction of the fibers and the degree of adhesion between the fibers and the polymer affect the degree of reinforcement (Nohrstrom *et al.* 2000; Saygili *et al.* 2003). Continuous unidirectional fibers give the highest strength and stiffness to the composite, but only in one direction, namely, in the direction of the fibers. Therefore, the reinforcing effect of unidirectional fibers is anisotropic in contrast to woven fibers, which reinforce the polymer in two directions and the composite has orthotropic mechanical properties (Kim and Watts 2004). If the fibers are orientated randomly as in a fiber mat, the mechanical properties are the same in all directions and the mechanical properties are isotropic (Kanie *et al.* 2000). Woven glass fiber has a suitable form as a reinforcing material for the so-called partial fiber reinforcement, because the woven glass fiber is a tape with various widths (Kanie *et al.* 2000). Furthermore, glass fiber is easy to cut with scissors but fray when bent excessively (Kanie *et al.* 2000). Polymer pre-impregnation eliminates this problem, but improper impregnation of the polymer matrix into the fiber bundle causes reduction in transverse strength of the polymer with glass fiber (Kanie *et al.* 2000). A lack of absorbed monomer liquid in the fiber bundle before polymerisation, can cause voids inside the test specimens (Kanie *et al.* 2000). The polymer pre-impregnated reinforcing glass fibers may considerably enhance flexural properties of the multiphase polymers, which is due to proper impregnation

of fibers with the polymer matrix (Hamza *et al.* 2004). Silanization of glass fibers also enhances the adhesion between the fibers and the polymer matrix, with increased fracture resistance and transverse strength of the polymers (Aydin *et al.* 2002).

1.3.4 Polyethylene fibers

Polyethylene, a naturally crystalline polymer, is drawn at temperatures below its melting point to produce a material of enhanced modulus in the axial direction (Braden *et al.* 1988). The Young's modulus (the index of the rigidity of the material) depends on the draw ratio, and moduli up to 60 GPa can readily be achieved for a draw ratio of 30 (Braden *et al.* 1988). To put these figures in perspective, polymethyl methacrylate is 3 GPa, which is considerably lower than the values possible with carbon fibres (250 GPa), but the polyethylene fibres are ductile and not as brittle (Braden *et al.* 1988). Their neutral colour, low density and known biological compatibility make them candidates for investigation as a reinforcing material for provisional restorative materials (Braden *et al.* 1988). They are drawn as monofilament fibres, but can also be woven into fabrics (Braden *et al.* 1988). Construct[®] (Kerr Corp, Orange, California, USA) consists of impregnated silanized plasma treated polyethylene fibers (Braden *et al.* 1988).

1.3.5 Metal wire reinforcement

The available literature on provisional restorative materials reinforced with metal wire is limited. Polyzois *et a*l (1995) examined the strength of heat-polymerized acrylic resin strips alone or in combination with metal wires.
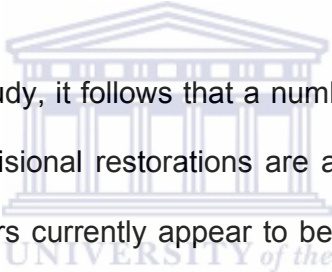
Their results indicated that metal wires might considerably enhance the strength of repaired denture acrylic resins.

Minami *et al* (2005) showed that specimens reinforced with 1.2 mm diameter stainless steel wires or Co-Cr-Ni wires resulted in significantly higher loads to fracture as compared to specimens without reinforcement. Reinforcement with glass fiber or round wire led to improved strength but a decrease in deflection at fracture, which in turn resulted in low toughness values (indicative of a weak material) (Minami *et al.* 2005). Despite these low toughness values, a more flexible and resilient product may withstand stresses more successfully without breaking, which is of clinical importance. Specimens reinforced with braided wire plate exhibited reduced strength when compared with specimens repaired with round wire as well as control specimens (Minami *et al.* 2005). This was attributed to the irregular shape of the braided wire, which may have inhibited proper penetration and complete wetting by the resin slurry and may have created weak points in areas of stress concentration (Minami *et al.* 2005).

1.3.6 Test methods

Researchers believe that fracture toughness is the best mechanical property measured to predict the wear and the fracture resistance of a restorative material (Higg *et al.* 2001). Different fracture toughness tests have been used to quantify the failure of dental materials. One such a test is the "Single-edge notch (SEN) three-point bending test". This test determines critical values of stress intensity (K_{IC}) where pre-cracked specimens of standard geometry are loaded until they break and the loads are used to calculate the toughness (Uctasli *et al.* 1995).

Haselton *et al* (2002) stated : "It is important to note that three-point flexural strength is only one of many behaviours in response to a particular stress and that strength is just one property of provisional crown materials. A strong material may possess other, less desirable characteristics such as tendency to stain, lack of polish ability, difficult manipulation or poor aesthetics. A provisional crown placed on a single anterior tooth will have different clinical requirements than a long-span provisional FPD. The clinician must be aware of all attributes of various materials and choose the provisional material and reinforcement method appropriate for each application".

From the literature study, it follows that a number of different techniques for reinforcement of provisional restorations are available, of which glass fiber and polyethylene fibers currently appear to be the most promising. As fiber reinforcement is likely to play an increasingly important role in the fabrication of provisional restorations, further investigations into the influence of fiber reinforcement on the fracture toughness of provisional materials are needed. Studies to determine the fractural strength of resins reinforced with steel wire, polyethylene fiber, or glass fiber, were previously carried out. However, none of them have compared all three of these strengtheners in a single study.

Research objectives

The objective of this study was:

To compare the fracture toughness of two provisional restorative materials without reinforcement, to their fracture toughness when reinforced with (a) steel wire, (b) polyethylene fiber and (c) glass fiber.

The null hypotheses tested were:

1. Without reinforcement, the two provisional restorative materials do not differ in fracture toughness.

2. Reinforcement with steel wire, polyethylene fiber, or glass fiber do not significantly alter the fracture toughness of the two provisional restorative materials.

Chapter 2

Materials and Methods

The method described in this chapter as a whole was based on a method described by Kim and Watts (2004). Two sample groups (52 samples/group) were prepared from the bis-acryl resin composite Protemp 3 Garant® and the polymethyl methacrylate resin, Coldpac® (MOTLOID, The Motloid Company, Chicago, Illinois, USA) for a three-point bending test conforming to the British Standard 5447 (1977). Each group was divided into four subgroups of thirteen specimens each to make provision for the three different types of reinforcement and one control group without any reinforcement (n=13) (Table 1).

Table 1: This table illustrates the two groups of provisional materials and the three types of reinforcement used (total sample size=104)

Reinforce-ment / Material	None	Glass fiber	Polyethylene fiber	Stainless steel wire
Coldpac®	13	13	13	13
Protemp 3 Garant®	13	13	13	13

As in Kim and Watts (2004), the overall external dimensions of the specimens were 3mm x 6mm x 25mm, excluding the positioning stops for the reinforcement materials.

A sharp central notch (3mm in length) was produced by inserting a straight-edged scalpel blade into a slot at mid-height in the mould; the slot extended down half the height to give A/W= 0.5 (Figure 1) (Kim and Watts 2004). The blade had a straight cutting edge honed on both sides, with a blade edge radius of less than 0.3µm (Solingen, England), forming a crack plane perpendicular to the specimen length. The mould used in this study could be split so that no force was required to remove the set specimens (Figure 2) from the mould (Figure 3).

The control groups (without any reinforcement) were fabricated according to the following procedure:

Protemp 3 Garant® was mixed using the auto mix gun system provided by the manufacturer. For Coldpac® a 0,58g powder to 0,25 ml liquid ratio was accurately measured and mixed, using a OHAUS precision standard scale (Model TS 400 D, Serial no 3630, OHAUS corporation, Florham Park, N.J, USA00) and a pipette (Pipetman, L 116956, Gilson, France) (Figure 4).

This mix resulted in a runny consistency so that the material could more easily be flowed into the mould, minimising air bubbles in the samples produced. After mixing, the materials were immediately transferred to the mould. The mould was slightly overfilled and the surface covered with a plastic matrix strip and a thick glass plate. Even though not described in the study by Kim and Watts (2004), a glass plate was pressed for 30 seconds until firm contact was established with the top surface of the template. This could be visualized through the glass plate. This was done in order to squeeze out the excess material, in an effort to minimize air bubbles and to produce samples of more uniform size. Specimens were left to polymerize at 23 ± 1°C as follows:

Coldpac® samples were left to polymerize for 20 min in the mould and then left an additional 10 min to bench cure (20 min was double the manufacturer's prescribed time needed for the material to set). Protemp 3 Garant® samples were left to polymerize for 10 min in the mould before removal and then left an additional 5 minutes to bench cure. Again, 10 min was double the time needed for the material to set according to manufacturer's instructions.

If to put into scale: B = 3 mm   S = 1,5 mm
                       W = 6 mm   T = 1,5 mm
                       A = 3 mm
                       L = 20 mm
                       X = 25 mm
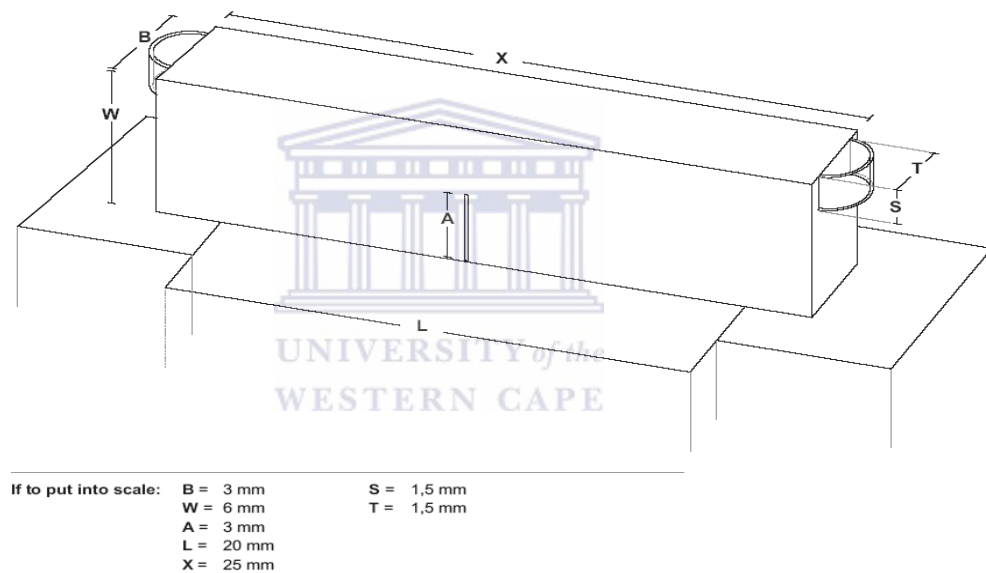
Figure 1: *Image of the notched specimen used in this study: A=depth of the notch, B=width, W=height, X=length of the specimen and L=the distance between the rests of the three-point bending apparatus (Image modified from the one used by Kim and Watts 2004.)*

The mould was then disassembled and the specimen removed. The blade was carefully removed and the specimens examined with a stereomicroscope (10X magnification) for the inclusion of voids or air bubbles. Those containing flaws were discarded. After removal from the mould, the edges of the specimens were finished with 1000 grit carbide

paper and specimen dimensions (width, height, and length) measured and recorded at three different points (Kim and Watts 2004).

For the reinforced test groups, all procedures were exactly the same as for the two un-reinforced resins, except for the insertion of the reinforcement material in the specimens. For the first group, everStick C&B fiber® (Stick Tech, Turku, Finland) was removed from the package and cut with sharp-edged scissors into 27mm-long pieces (Figure 5). The mould was filled up to level C (Figure 2) with one of the resins and the light cured fibers placed parallel to the long axis of the specimen, into the un-cured material. The fibers were polymerised by irradiating three different areas (centre, left and right) with a light-curing unit (Megalux CS, Megadenta, Radeberg, Germany) for 40 seconds each. The two lateral stops in the mould (Figure 3) ensured that the different reinforcing materials were inserted at the same position in all specimens. Resin was then added to fill the mould as described before.

For the second group Construct® reinforcing braid was used (Figure 6). It was cut into strips of 27mm and impregnated with resin (according to the manufacturer's instructions), cured and placed in the mould exactly as described for the everStick C&B fiber®. The third group was similarly reinforced, but instead of using fibers, a 27mm long orthodontic stainless steel wire (1mm in diameter) (KC Smith & Co., Monmouth, United Kingdom) was imbedded into the unset resin before the mould was filled.

The specimens were stored in distilled water at 37 ^{0}C in an oven (Memmert, 854, Schwabach, West Germany) for 24 hours before testing. Thereafter, they were tested in three-point bending using a Zwick Universal Testing Machine (Model 1446, Zwick, Ulm, Germany). The specimens were placed on the supports of the three-point bending apparatus with a fixed span width

of 20mm (Figure 7). Mechanical loading was applied on the centre of each specimen at 90 degrees to the specimen axis through a stainless steel rod attached to the Zwick Universal Testing Machine. A load was applied using a 0.5Kg loading cell at a crosshead speed of 1 mm/s until the specimen fractured. The fracture lines were also inspected with a stereomicroscope (10X magnification) for the inclusion of air bubbles and voids. The results of the ones containing flaws were ignored and a replacement sample was used. Peak load to fracture, and specimen deflection (recorded as load/deflection curves) were recorded and fracture toughness (K_{IC} measured in $MNm^{-1.5}$) was calculated using the following equation:

$$(K_{IC}) = (3PL/BW^{3/2})Y$$

where P=peak load at fracture; L=length; B=width; W=height; and Y=calibration function ($1,93 \{A/W\}^{1/2} - 3,07 \{A/W\}^{3/2} + 14,53 \{A/W\}^{5/2} - 25,11 \{A/W\}^{7/2} + 25,80 \{A/W\}^{9/2}$).

The two different resins (controls) and the three reinforcements of each resin (six test groups) resulted in eight different combinations of resins and reinforcements. The mean and standard deviations for each test group were calculated. The medians of the values for these eight different combinations of materials containing two controls of un-reinforced (polymethyl methacrylate and bis-acryl) resin were compared (pair wise and otherwise) by means of non-parametric analysis of variance ($p<0.05$). The results were then compared for statistical significant differences (Kruskal-Wallis) and summarized taking into account the Bonferroni adaptation for multiple comparisons.
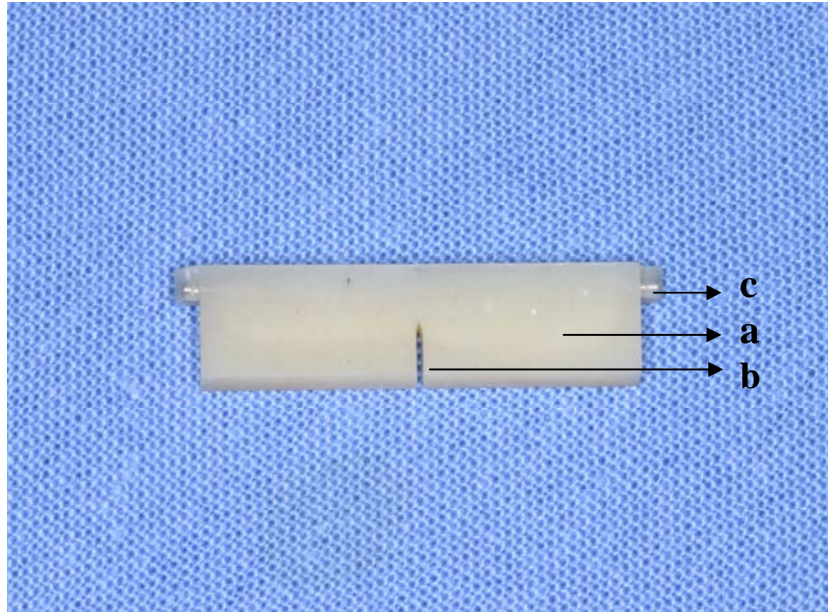
Figure 2: *Specimen (a) showing the central groove (b) and the position of the reinforcement (c)*
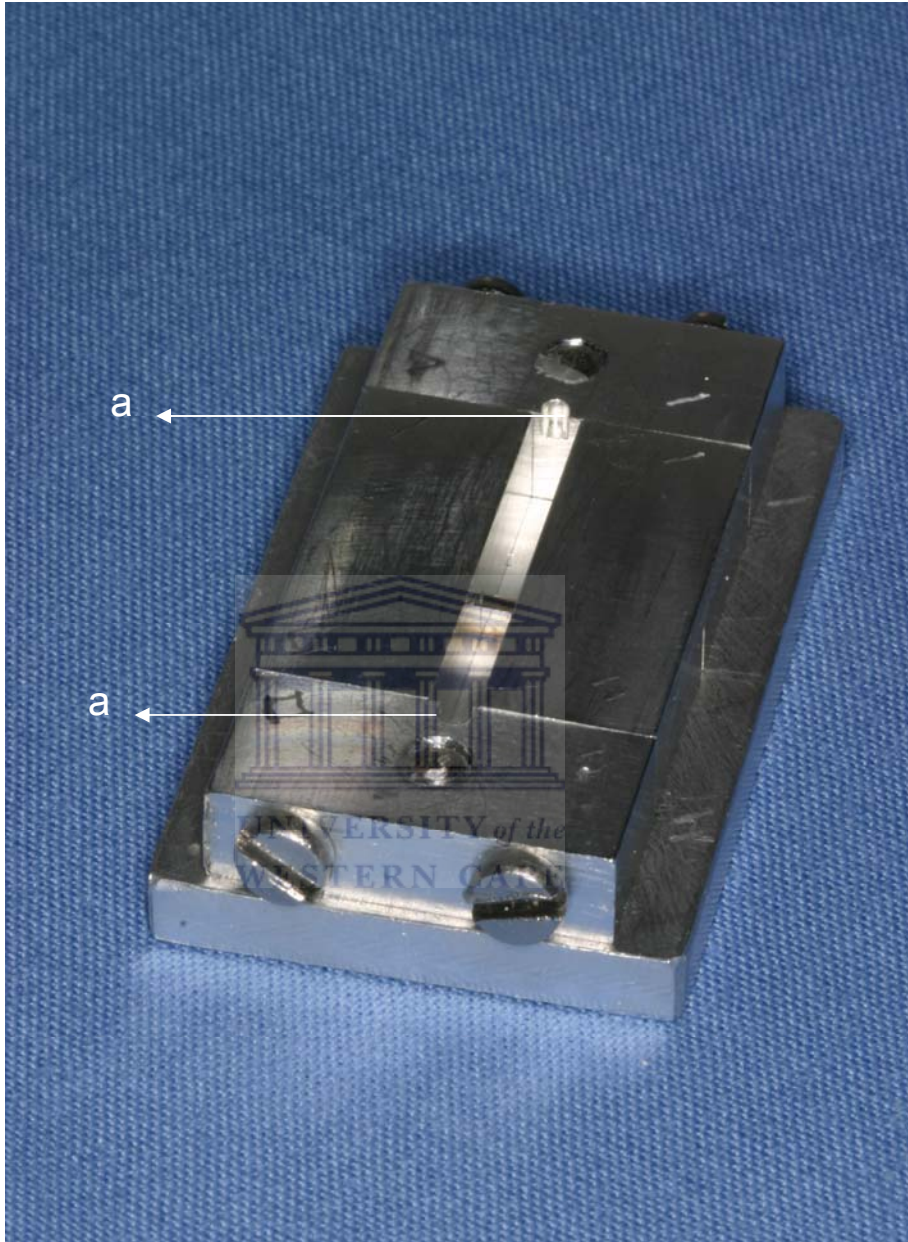
Figure 3: *Stainless steel mold, used to fabricate the samples, showing the notches (a) where the reinforcement was placed*

Figure 4: *OHAUS precision standard scale Model TS 400 D (a) and a pipette (b)*
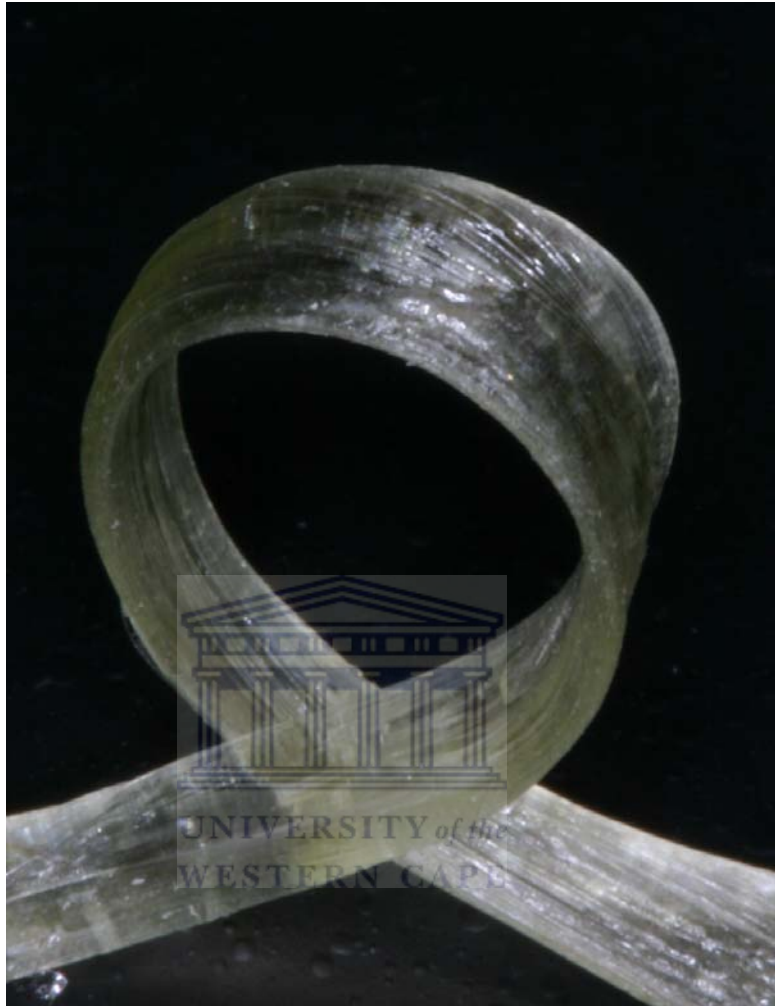
Figure 5: *everStick fiber® glass braid*
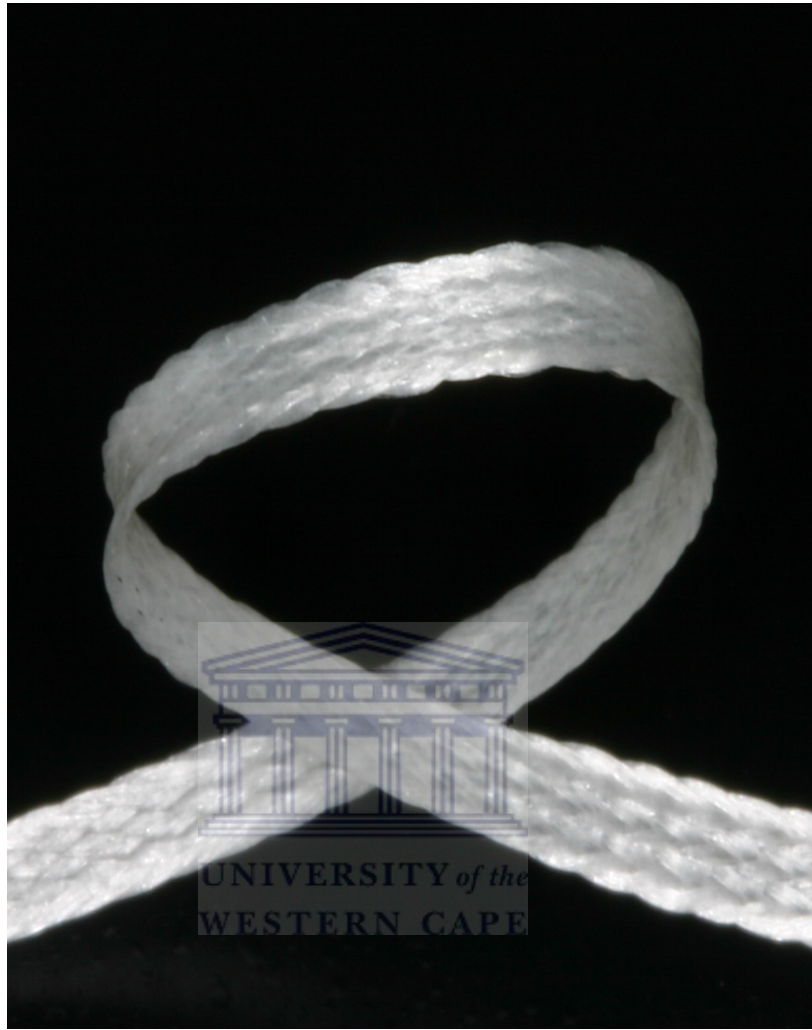
Figure 6: *Construct® fiber braid*

Figure 7: *Showing specimen (a) on the three-point bending apparatus (b)*

Chapter 3

Results

The results from this experiment reflect:

1) Fracture toughness of the two provisional resins Protemp 3 Garant® and Coldpac® (controls).

2) Fracture toughness of the same two resins reinforced with three different reinforcing materials (steel wire, glass fibers and polyethylene fibers).

The variability and descriptive statistics for the fracture toughness values (K_{IC}) calculated for the different measurements are illustrated by means of box plots (Figures 8-13) and a supplementary violin plot (Figure14).

The relationship between the calculated measurement, fracture toughness, and a categorical variable, the material used, is best visualized by side-by-side (parallel) Box-and-Whisker plots. Box plots have the advantage over the other types of plots that they highlight outliers (unusual measurements that lie well away from the central points on the plot).

The bottom boundary of the yellow box is the 25th percentile of the sample, the middle line the median (50th percentile) and the top boundary of the yellow box the 75th percentile. The yellow box contains 50% of the particular sample's observations. Unusual values (or outliers) in the sample are displayed by means of coloured green or red dots. These values are unusual with respect to the distance from the median (measured in terms of the inter-quartile range).

The violin plot, in which a "density trace" is fitted to the points, estimates the underlying distribution function of the values. In a violin plot, both the density trace and its mirror reflection are shown in the plot.

In the section below, control Coldpac® and control Protemp 3 Garant® are compared and thereafter the fracture toughness of these two materials are compared with wire, polyethylene fiber and glass fiber added to both.

Table 2: Table of descriptive statistics listing fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of the control materials Coldpac® and Protemp 3 Garant®, with a sample size of 13 in each case

Data	Control Coldpac®	Control Protemp 3 Garant®
Minimum	25.89	30.86
Median	27.89	31.17
Average of Stress	29.07	35.32
Standard Deviation	2.63	2.38
Interquartile Range	5.25	1.91
Maximum	33.79	40.72

In the Table above both averages were higher than the medians, indicating that there was some skewness towards the higher values. The median of Protemp 3 Garant® was higher than that of Coldpac® and this is confirmed by a Kruskal-Wallis test ($p<0.01$). It could be observed that the two standard

deviations are approximately equal, but that the interquartile range of Protemp 3 Garant® (1.91) was much smaller than that of Coldpac® (5.25).
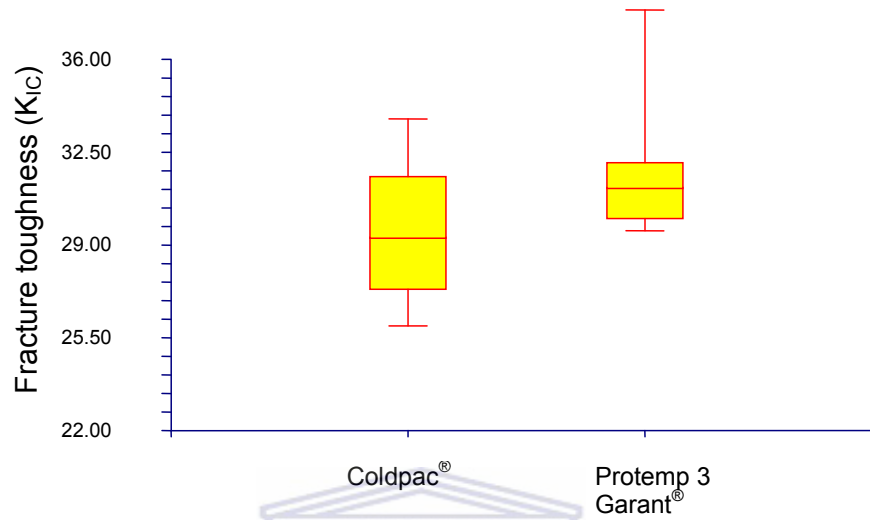
Figure 8: *Side-by-side Box-and-Whisker Plot of fracture toughness (K_{IC} measured in $MNm^{-1.5}$) comparing* **control** *Coldpac® to* **control** *Protemp 3 Garant®*

Table 3: Table of descriptive statistics listing fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of Coldpac® and Protemp 3 Garant® reinforced with wire, with a sample size of 13 in each case

Data	Coldpac® & wire	Protemp 3 Garant® & wire
Minimum	32.47	40.93
Median	39.00	44.02
Average of Stress	42.74	45.97
Standard Deviation	5.02	3.21
Interquartile Range	5.85	3.74
Maximum	50.79	53.11

The standard deviation of Coldpac® and wire was somewhat larger than the SD of Protemp 3 Garant® and wire, but it was not significantly different. The same was true for the interquartile range. The median of Protemp 3 Garant® was larger than that of Coldpac® and this is confirmed by a Kruskal-Wallis test (p<0.005).

Figure 9: *Side-by-side Box-and-Whisker Plot demonstrating the difference in fracture toughness (K_{IC} measured in $MNm^{-1.5}$) comparing Coldpac® to Protemp 3 Garant®, reinforced with wire*

Table 4: Table of descriptive statistics listing fracture toughness (K_{IC} measured in MNm$^{-1.5}$) of Coldpac® and Protemp 3 Garant® reinforced with polyethylene fiber, with a sample size of 13 in each case

Data	Coldpac® & poly-ethylene fiber	Protemp 3 Garant® & poly-ethylene fiber
Minimum	24.59	22.60
Median	25.82	33.10
Average of Stress	29.79	35.77
Standard Deviation	3.87	4.46
Interquartile Range	2.75	3.56
Maximum	38.22	41.41

The stress measurements of the two materials reinforced with polyethylene fiber were skewed towards the higher values as is evident from comparing the average and the medians. The level of the fracture toughness median of Coldpac® reinforced with polyethylene fiber was higher than the median level of Protemp 3 Garant® reinforced with polyethylene fiber ($p < 0.001$).

Figure 10*: Side-by-side Box-and-Whisker Plot demonstrating the difference in fracture toughness (K_{IC}measured in $MNm^{-1.5}$) of Coldpac® and Protemp 3 Garant® reinforced with polyethylene fibers*

Table 5: Table of descriptive statistics listing fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of Coldpac® and Protemp 3 Garant® reinforced with glass fiber with a sample size of 13 in each case

Data	Coldpac® & glass fiber	Protemp 3 Garant® & glass fiber
Minimum	33.27	40.43
Median	34.44	42.31
Average of Stress	40.01	46.75
Standard Deviation	8.85	3.88
Interquartile Range	5.94	3.31
Maximum	62.76	52.41

The standard deviation and interquartile range of Coldpac® were larger than

that of Protemp 3 Garant®, but not significantly different. The medians of

Coldpac® and Protemp 3 Garant®, both reinforced with glass fiber, were

different (p<0.01).
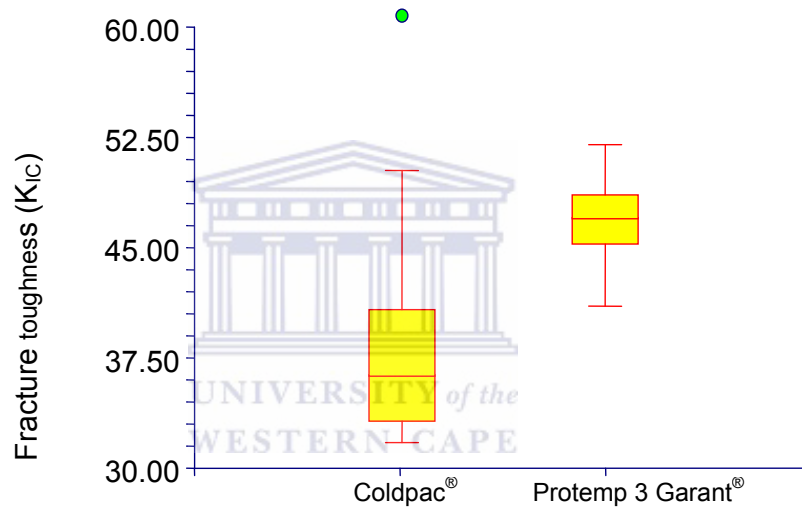
Figure 11: *Side-by-side Box-and-Whisker Plot demonstrating the difference in fracture toughness (K_{IC} measured in MNm$^{-1.5}$) of Coldpac® and Protemp 3 Garant® reinforced with glass fiber*

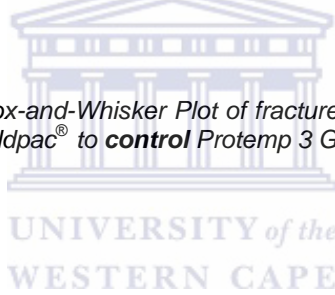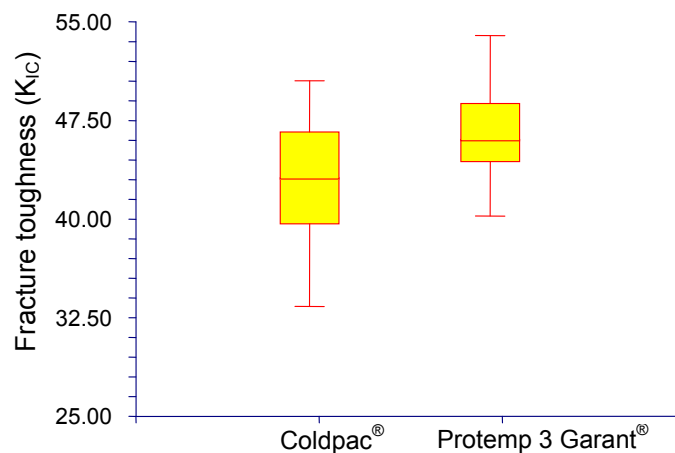**Table 6**: Table of descriptive statistics listing fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of Coldpac® reinforced with wire, polyethylene fiber and glass fiber, respectively, with a sample size of 13 in each case

Data	Control Coldpac®	Coldpac® & wire	Coldpac® & polyethylene fiber	Coldpac® & glass fiber
Minimum	25.89	32.47	24.59	33.27
Median	27.89	39.00	25.82	34.44
Average of Stress	29.07	42.74	29.79	40.01
Standard Deviation	2.63	5.02	3.87	8.85
Interquartile Range	5.25	5.85	2.75	5.94
Maximum	33.79	50.79	38.22	62.76

Coldpac® and glass fiber displayed the largest standard deviation and interquartile range of all four material combinations. The differences between the standard deviation's of the four materials were not statistically significantly different (p>0.05). The highest and lowest fracture toughness medians were highly significantly different (p<0.000001), with Coldpac® reinforced with wire exhibiting the highest median and Coldpac® reinforced with polyethylene fiber, the lowest median.
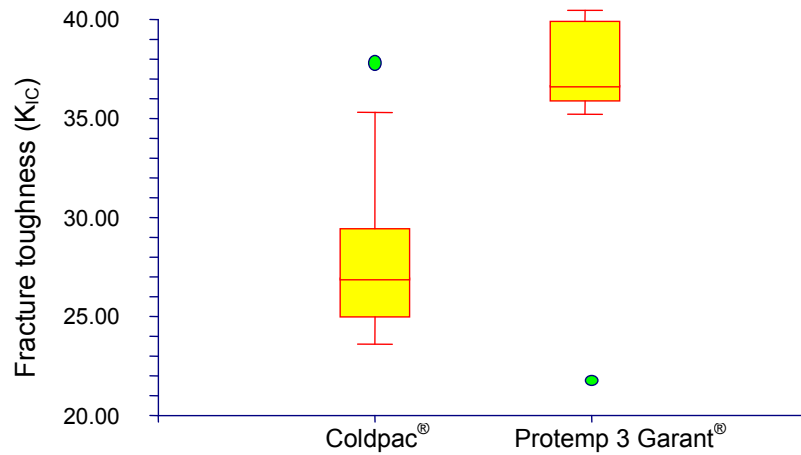
Figure 12: *Combined box plots demonstrating the difference in fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of Coldpac® on its own and reinforced with the three different reinforcement materials*

The side-by-side box plot above shows a high outlier value for the measurements (K_{IC}) of Coldpac® reinforced with glass fiber. Another outlier value occurred for Coldpac® reinforced with polyethylene fiber.
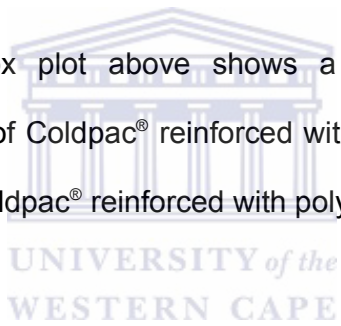
Table 7: Table of descriptive statistics listing fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of Protemp 3 Garant® reinforced with wire, polyethylene fiber and glass fiber, respectively, with a sample size of 13 in each case

Data	Control Protemp 3 Garant®	Protemp 3 Garant® & wire	Protemp 3 Garant® & polyethylene fiber	Protemp 3 Garant® & glass fiber
Minimum	30.86	40.93	22.60	40.43
Median	31.17	44.02	33.10	42.31
Average of Stress	35.32	45.97	35.77	46.75
Standard Deviation	2.38	3.21	4.46	3.88
Interquartile Range	1.91	3.74	3.56	3.31
Maximum	40.72	53.11	41.41	52.41

Protemp 3 Garant® and polyethylene fiber reinforcement displayed the largest standard deviation of all four material combinations containing Protemp 3 Garant®. The differences between the standard deviations of the four materials were not statistically significantly different ($p > 0.05$). The samples containing only Protemp 3 Garant® displayed the smallest standard deviation and interquartile range. However, the highest and lowest fracture toughness medians were highly significantly different ($p < 0.000001$), with Protemp 3 Garant® reinforced with wire exhibiting the highest median and Protemp 3 Garant® reinforced with glass fiber, the highest average. The fracture toughness as measured by the medians and averages, differs slightly for these two materials, Protemp 3 Garant® reinforced with wire and Protemp 3 Garant® reinforced with glass fiber.
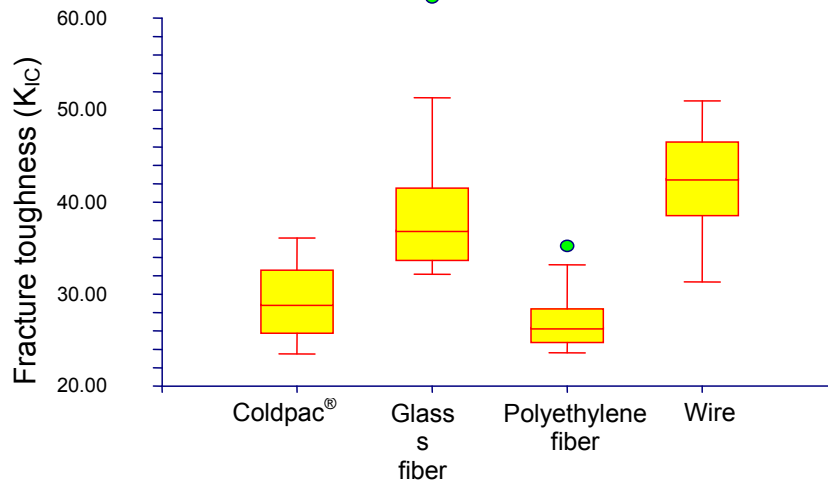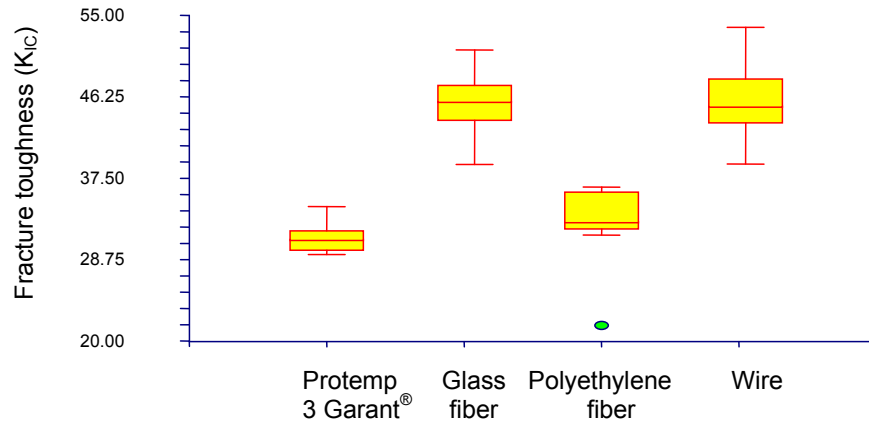
Figure 13: *Combined box plots demonstrating the difference in fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of Protemp 3 Garant® resin on its own and reinforced with the three different reinforcement materials*

Table 8: Descriptive statistics listing fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of the eight groups, with a sample size of 13 in each case

	Coldpac®				Protemp 3 Garant®				all K_{IC} values
	control	wire	PE	glass	control	wire	PE	glass	
Min	25.89	32.47	24.59	33.27	30.86	40.93	22.60	40.43	**22.60**
Med	27.89	39.00	25.82	34.44	31.17	44.02	33.10	42.31	
Av	29.07	42.74	29.79	40.01	35.32	45.97	35.77	46.75	**38.18**
SD	2.63	5.02	3.87	8.85	2.38	3.21	4.46	3.88	**7.85**
IR	5.25	5.85	2.75	5.94	1.91	3.74	3.56	3.31	
Max	33.79	50.79	38.22	62.76	40.72	53.11	41.41	52.41	**62.76**

PE = polyethylene fiber, glass = glass fiber, Min = minimum, Med = median, Av = average of stress, SD = standard deviation, IR = interquartile range, Max = maximum.

Protemp 3 Garant® reinforced with wire exhibited the largest median (44.02), Protemp 3 Garant® reinforced with glass fiber the second largest (42.31), and Coldpac® reinforced with wire (39.00) the third largest. The same three material combinations make up the three largest averages, namely Protemp 3 Garant® reinforced with wire (45.97; second highest), Protemp 3 Garant® reinforced with glass fiber (46.75; highest), and Coldpac® reinforced with wire (42.74; third highest). It is important to note that Coldpac® reinforced with polyethylene fiber resulted in the lowest median fracture toughness (25.82) and it is even weaker than control Coldpac® (27.89; the second smallest).
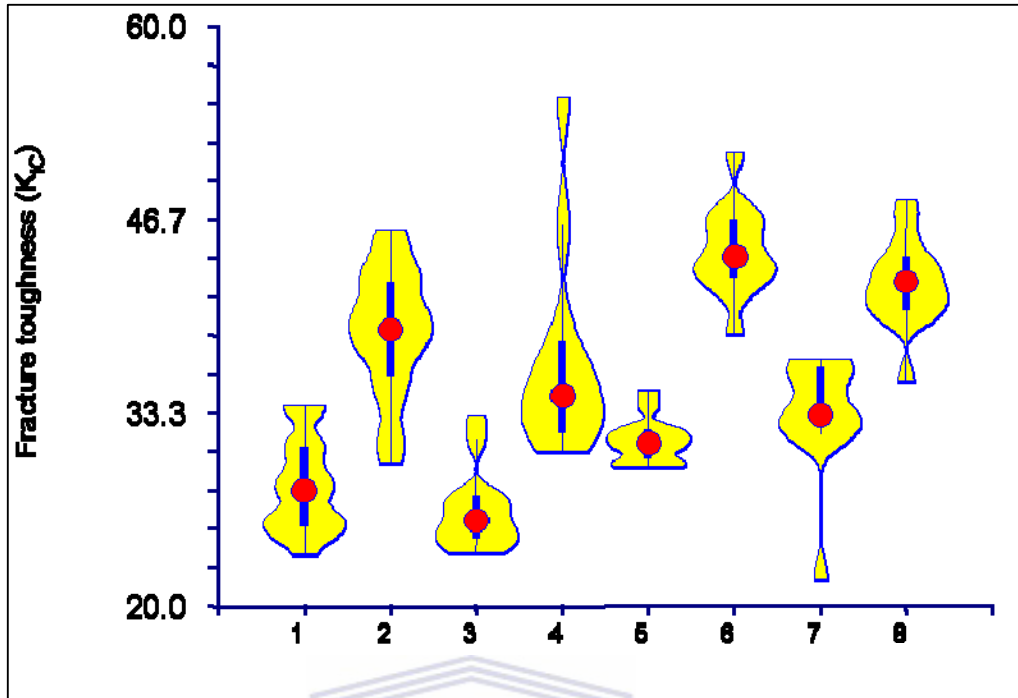
Figure 14: *Combined Side-by-side Violin plot demonstrating the difference in fracture toughness (K_{IC} measured in $MNm^{-1.5}$) of the two resins, without reinforcement and with the three different reinforcement materials*

Legend:

1	Coldpac® as control	5	Protemp 3 Garant® as control
2	Coldpac® + 1mm stainless steel wire	6	Protemp 3 Garant® + 1mm stainless steel wire
3	Coldpac® + polyethylene fibers	7	Protemp 3 Garant®+ polyethylene fibers
4	Coldpac® + glass fibers	8	Protemp 3 Garant® + glass fibers

Chapter 4

Discussion

Provisional crowns and FPDs are essential components of prosthodontic treatment. Provisional restorations must satisfy biologic and aesthetic needs as well as mechanical requirements such as resistance to functional loads, resistance to removal forces and maintenance of abutment alignment. The fracture toughness and flexural strength of provisional materials is important, particularly when the patient must use the provisional restoration for an extended period, when the patient exhibits parafunctional habits, or when a long-span prosthesis is planned.

This study compared the fracture toughness of two materials commonly used to fabricate provisional restorations namely Coldpac®, a polymethyl methacrylate and Protemp 3 Garant®, a bis-acryl composite. It also compared the fracture toughness of the two materials when reinforced with 1mm stainless steel wire, glass fibers (everStick C&B fiber®) and polyethylene fibers (Construct® reinforcing braid).

The null hypotheses tested were that the two provisional restorative materials don't differ in fracture toughness and that reinforcement with steel wire, polyethylene fiber, or glass fiber do not raise the fracture toughness of the two provisional restorative materials.

The method that was used to determine the fracture toughness of the specimens was a three-point bending test based on a method described by Kim and Watts (2004). The mould was specially adapted to ensure that the

reinforcement materials were placed in the same position in all the specimens. To do this, a 1.5mm deep notch was included on each side of the mould (a in Figure 3). This is important, as the position of the reinforcement material in the specimen will have an effect on the fracture toughness of the material being tested (Lassila and Vallittu 2004; Nohrstrom *et al.* 2000). Although laboratory fracture toughness values under static loading may not reflect intra-oral conditions, these values are nevertheless helpful in comparing materials under controlled situations, and may be useful in predicting the clinical performance of the materials tested.

Usually, during fabrication of the specimens, it is very difficult to eliminate all flaws within the specimens. These flaws may have a direct effect on flexural strength values and therefore, researchers believe that fracture toughness is the best mechanical property to measure for materials used to fabricate provisional FPDs (Hamza *et al.* 2004; Higg *et al.* 2001).

Within the limits of this experiment, the results indicated that the polymethyl methacrylate provisional material, Coldpac®, has a significantly (p<0.01) lower median fracture toughness (K_{IC}) (27.89 MNm$^{-1.5}$) when compared to the bis-acryl composite, Protemp 3 Garant® (31.17 MNm$^{-1.5}$) (Figure 13). This finding is in agreement with the results of Lang *et al* (2003) who reported the highest strength values for Protemp 3 Garant® compared to polymethyl methacrylate materials when interim FPDs were constructed from these materials, artificially aged and tested for fracture resistance. Higher flexural strengths for bis-acryl composite resins compared to polymethyl methacrylates were also reported by Haselton *et al* (2002). However, Koumjian and Nimmo (1990) as well as Osman and Owen (1993) reported higher flexural strength values for polymethyl methacrylate materials

compared to bis-acryl composite resins. A possible explanation for these findings could be that, since then, Protemp® has been modified and marketed as Protemp 3 Garant® and used in this experiment.   The modifications include a newly developed monomer system, not with the rigid intermediate chain characteristic of some bis-acryl homologues, but with a somewhat flexible chain in comparison to other synthetic resins. According to the manufacturer, this attribute allows a balance between high mechanical strength and limited elasticity of the composite material. This was confirmed by a study done in 2002 by Haselton *et al* who reported a significant increase in the flexural strength of Protemp 3 Garant® compared to its predecessor.

Another reason for the lower fracture toughness values for Coldpac® in this study could be attributed to the fact that the material was not mixed according to the manufacturer's instructions as this would have resulted in a very stiff consistency. Instead, Coldpac® was mixed 0,58g powder to 0,25 ml liquid ratio. This mix resulted in a runny consistency so that the material could more easily be flowed into the mould, minimising air bubbles in the samples produced. It is possible that this action could have weakened the material. However, this is not important for the consideration of hypothesis 2 as care was taken that the mix was the same for all the Coldpac® samples used to fabricate the reinforced specimens.

Many investigators have confirmed the reinforcing effect of fibers and steel wire on different polymer types (Aydin *et al.* 2002; Dyer *et al.* 2005; Hamza *et al.* 2004; Kim and Watts 2004; Van Ramos *et al.* 1996; Samadzadeh *et al.* 1997; Stiesch-Scholz *et al.* 2005; Vallittu 1998). In this study it was found that the glass fibers significantly increased the fracture toughness of both

types of provisional materials, (p<0.01 for Coldpac® and p<0.001 for Protemp 3 Garant®). The stainless steel wire significantly increased the fracture toughness of both provisional FPD materials, (p<0.001 for Coldpac® and p<0.0001 for Protemp 3 Garant®). However, polyethylene fibers did not increase the fracture toughness of the two materials significantly, (p>0.05 for Coldpac® and p>0.10 for Protemp 3 Garant®). Hypothesis 2, stating that reinforcements with steel wire, polyethylene fiber, or glass fiber do not raise the fracture toughness of the two provisional restorative materials was therefore only partially rejected for the stainless steel wire and for the glass fibers (these two reinforcements strengthened fracture toughness), but accepted for polyethylene fibers (did not improve fracture toughness).

The reinforcing ability of glass fibers and polyethylene fibers was explained by Nohrstrom and co-workers (2000). They stated that transfer of stress takes place from the weaker polymer matrix to the fibers with a higher tensile strength. This means that the better the adhesion between the fibers and the matrix of the resin, the greater the strengthening effect.

The surprising finding that polyethylene fibers did in fact lower the fracture toughness of the Coldpac® material could possibly be explained by the fact that Coldpac® was not mixed according to the manufacturer's instructions. Protemp 3 Garant® was mixed using the auto mix gun system provided by the manufacturer, but Coldpac® was mixed 0,58g powder to 0,25 ml liquid ratio, resulting in a mixture of lower viscosity. Although a reduced viscosity should theoretically improve impregnation of fibers into the resin, it was shown by Vallittu (1999) that a higher proportion of monomer liquid in the mixture would increase the polymerisation shrinkage of the resin applied to the fiber bundle. This higher polymerisation shrinkage will cause a slit

between the fibers and the polymer matrix. This improper degree of impregnation will also increase the water sorption that might lead to a detrimental hydrolytic effect and a resulting decrease in mechanical properties of the reinforced resin (Vallittu 1999). In contrast to this finding Van Ramos, *et al* (1996) reported a significant increase in the fracture strength of polymethyl methacrylate reinforced with plasma-treated polyethylene fiber (Ribbond®). Noteworthy of their experiment is the fact that the polymethyl methacrylate was mixed at a 2.5:1 polymer-to-monomer ratio, whereas the ratio of the mix in the current study was 2.32:1.

This experiment also determined the reinforcement capability of the three different reinforcement materials and was statistically evaluated, comparing their ability to reinforce the two different resins. It was found that all three reinforcements increased the fracture toughness of Protemp 3 Garant® significantly more than for Coldpac® (glass fiber p<0.005; polyethylene fiber p< 0.005; wire p<0.005). This finding is supported by Hamza *et al* (2004) who reported that Fibrestick® as well as Construct® increased the fracture toughness of FPDs compared to the un-reinforced controls. Samadzadeh *et al,* (1997) using Coldpac® (polymethyl methacrylate) and Provipoint DC® (bis-acryl) also reported a higher increase of load to fracture of the bis-acryl (65.59 kg) compared to the polymethyl methacrylate (53.46 kg), although Provipoint DC® had a lower fracture strength (46.59 kg) compared to Coldpac® (49.86 kg) when un-reinforced.

Combined reports for Coldpac® and Protemp 3 Garant® reinforced with the different reinforcing materials were also compiled. The results in Chapter 3 show that the 1mm stainless steel wire increased the fracture toughness of Coldpac® significantly more than reinforcement with polyethylene

(p<0.0001), but not significantly more than reinforcement with glass fiber (p>0.10). This finding is supported by the results reported by Vallitu, *et al* (1995) although they used a heat-cured polymethyl methacrylate, (ProBase Hot®) and measured impact strength.

Glass fiber increased the fracture toughness of Coldpac® significantly more than polyethylene (p<0.0001). This is in agreement with other authors (Hamza *et al.* 2004; Kim and Watts 2004; Vallittu 1999). Although the polyethylene fibers reduced the fracture toughness of the Coldpac® control, it was not significant (p>0.10).

From Chapter 3 it is clear that the 1mm stainless steel wire increased the fracture toughness of Protemp 3 Garant® significantly more than when reinforcement with polyethylene fiber (p<0.0001), but again not significantly more than glass fiber (p>0.10). Glass fiber increased the fracture toughness of Protemp 3 Garant® significantly more than polyethylene fiber (p<0.005). This finding is supported by Hamza *et al* (2004) using Ribbond® and Fibrestick® with Jet® as the polymethyl methacrylate and Temphase® as the bis-acryl.

Unlike the negative effect on the fracture toughness of Coldpac® control, polyethylene fibers did reinforce Protemp 3 Garant® control, but not significantly (p>0.10). Kolbeck *et al* (2002) also reported that the reinforcing effect of glass fibers was more effective than that of polyethylene fibers. They attribute this to the difficulty of obtaining good adhesion between the polyethylene fibers and the resin matrix.

Very few, if any, studies were done that include all three of these reinforcement materials. Thus, it was not possible to compare the results of the wire reinforcement with glass fiber and polyethylene fibers comprehensively.

Limitations of the study:

1. Coldpac® mixture:

   A possible limitation of this study is the powder and liquid ratio of the mixture that was used. This has been fully discussed on page 38 and 39. A pilot study could have been of value to determine the optimum powder/liquid ratio in order to minimize the inclusion of air bubbles while obtaining the maximum physical properties.

2. Water storage:

   In this experiment, it was decided not to store the specimens in water for periods longer than 24 hours before testing for fracture toughness (see Materials and Methods section). This decision was based on the results of a study done by Kim and Watts in 2004 where they evaluated the effect of water storage (1, 7, 30 and 60 days) on un-reinforced as well as reinforced (with glass fiber) polymethyl methacrylate and bis-acryl materials. They could demonstrate no significant decrease in K_{IC} values of the reinforced materials after 2 months of water storage compared with the values at day 1 (Kim and Watts 2004).

3. The use of 1mm stainless steel wire:

   The inclusion of the wire in this experiment may not be of clinical relevance as the wire was covered by at least 1mm of resin on all

sides (this was done in order to standardize the samples with those of the other reinforcement materials). This scenario may not always be possible in the clinical situation. However, it serves an important purpose as a standard for measuring the other reinforcement materials and provides the clinician with an additional and cheaper option in posterior areas of the mouth where aesthetics is not important and enough space is available.

Possible Further Studies

1. Investigators reported extensively on the reinforcement value of stainless steel (1mm) on bis-acryl resins used to fabricate partial and full denture bases (Vallittu 1996; Vallittu 1993; Vallittu and Lassila 1992). However, apart from this one, no single study was found that compared the fracture toughness of provisional FPDs reinforced with stainless steel wire, glass fibers or polyethylene fibers.

Comparing fracture toughness using different diameters of stainless steel wire that is placed in different positions in samples that represent provisional FPDs, clinically, would result in valuable information.

2. Further studies on the polymethyl methacrylate powder to liquid ratio, using a specific reinforcement material, will also supply valuable information to clinicians on the flexural-, tensile-, and impact strengths as well as the fracture toughness of the polymethyl methacrylate resins and may influence their decision on the choice of materials that can be used in the fabrication of provisional FPDs.

Chapter 5

Conclusions and recommendations

Within the limitations of this study, it was concluded that:

1. Coldpac® control has less fracture toughness than Protemp 3 Garant® control ($p < 0.01$).

2. All three reinforcements increased the fracture toughness of Protemp 3 Garant® significantly more than for Coldpac® (glass fiber - $p < 0.005$; polyethylene fiber - $p < 0.001$; steel wire - $p < 0.005$).

3. The glass fibers significantly increased the fracture toughness of both types of provisional materials ($p < 0.005$ for Coldpac® and $p < 0.0001$ for Protemp 3 Garant®). The stainless steel wire also significantly increased the fracture toughness of both provisional materials ($p < 0.0001$ for Coldpac® and $p < 0.0001$ for Protemp 3 Garant®). However, it was found that the polyethylene fibers did **not** increase the fracture toughness of the two provisional materials significantly, ($p > 0.10$ for Coldpac® and $p > 0.10$ for Protemp 3 Garant®).

4. For Coldpac® the 1mm stainless steel wire increased the fracture toughness significantly more than the reinforcement with polyethylene fiber ($p < 0.0001$), but **not** significantly more than reinforcement with glass fiber ($p > 0.10$). Glass fiber reinforcement increased the fracture toughness of Coldpac® significantly more than that of polyethylene fibers ($p < 0.0001$), but polyethylene fibers weakened the polymethyl methacrylate material, although **not** significantly ($p > 0.10$).

5. For Protemp 3 Garant® the 1mm stainless steel wire increased the fracture toughness significantly more than reinforcement with polyethylene fiber ($p<0.0001$), but again **not** significantly more than glass fiber ($p>0.10$). Glass fiber increased the fracture toughness of Protemp 3 Garant® significantly more than polyethylene fiber ($p<0.005$).

To summarise:

1. Coldpac® has less fracture toughness than Protemp 3 Garant®.

2. A 1 mm stainless steel wire and everStick C&B fiber® increases the fracture toughness of Protemp 3 Garant® and Coldpac®, while Construct® reinforcing braid does not.

3. Reinforcement of both types of resins with stainless steel wire, provides the highest fracture strength to the clinician, but aesthetics and availability of space may restrict its use.

4. Where aesthetics is of concern, everStick C&B fibers® seems to be the most successful when reinforcing Protemp 3 Garant® as well as Coldpac®.

5. The mixing ratio of polymethyl methacrylate materials may be of critical importance in the manufacturing of provisional fixed partial dentures.

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