THE DEVELOPMENT AND OPTIMIZATION OF A COSMETIC FORMULATION THAT FACILITATES THE PROCESS OF DETANGLING BRAIDS FROM AFRICAN HAIR.

By

KWEZIKAZI MKENTANE

STUDENT NUMBER: 20537842

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Supervisor: Dr NM Vorster
Co-Supervisor: Mr J Bosma

CONFIDENTIALITY

The information pertaining to the formulation contained in this dissertation is regarded as being of a confidential nature and is protected under provisional patent nr 2011**/04055 for: HAIR BRAID REMOVER FORMULATION**

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Declaration

I hereby declare that this dissertation is my own original work. It is being submitted for the degree of Magister Scientiae (Chemistry) at the Nelson Mandela Metropolitan University. It has not been submitted before for any degree or examination in any other University or Technikon.

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EXECUTIVE SUMMARY

A large number of people throughout the world have naturally kinky hair that may be very difficult to manage. These people often subject their hair to vigorous and harsh treatment processes in order to straighten it and hence make it more manageable. Hair braiding is a popular and fashionable trend amongst many people, in particular people of African descent. Braided hairstyles serve to preserve hair and protect it, and to give it time to rejuvenate after a period of harsh treatment. During the braiding process synthetic hair is attached to natural hair by weaving a length of the natural hair into one end of each braid. Other materials like wool or cotton may be use used to achieve different hairstyles and textures. Several strands of natural hair are used to secure each braid. The braids are normally left intact for a number of weeks or even months. Although braiding is a helpful African hair grooming practice, the process of taking down or detangling the braids is labor intensive and entails each braid being cut just below where the natural hair ceases and the natural hair being untangled from the braid using a safety pin, a needle or a fine toothed comb. The labor and long hours required to detangle braided hairstyles often results in braid wearers frustratingly pulling on their braided hair. This behavior inevitably destroys the hair follicle and leaves the hair damaged. According to a study conducted by the University of Cape Town's dermatology department, braiding may be the root cause of traction alopecia (TA) amongst braid wearers. Traction alopecia is a form of alopecia, or gradual hair loss that is caused primarily by excessive pulling forces applied to the hair.

The purpose of this current study was to investigate the factors, other than braid tightness, that affect the way and ease with which braids are detangled from the human hair. The study hypothesized that frictional forces present in braided hair were amongst these factors. It was hypothesized that introducing a lubricating formulation in the braids would allow for easier braid detangling. In order to decrease the prevalence of traction alopecia from braided hair, two hair strengthening actives were included in the test formulation. The study investigated the effects of the test formulations on braid detangling, hair friction and on the tensile strength of human hair. The study found that the method used did not pick up any significant differences between the braid detangling forces of treated braids when compared to the braid detangling forces of untreated hair. The same method used to measure braid detangling forces was able to show that there are variations in the braid detangling forces of different sections along the braid length. The method to measure braid detangling was based on the principles of hair combability measurements.

The study also found that although the method used to measure braid detangling forces was unsuccessful in picking up significant differences in braid detangling forces of treated hair and untreated hair, the method used to measure the frictional forces of human hair showed that the frictional forces of hair treated with test formulations were significantly different than that of untreated hair. The method used to measure frictional forces was based on the capstan approach. The Capstan method measures the forces required to slide a weighted hair fibre over a curved surface of reference material. The interaction between the weighted fibre and the reference material simulates the movement of hair out of a braid ensemble in the braid detangling process.

The optimum mixture with the minimum coefficient of friction, predicted a coefficient of friction of 0.61 ± 0.04. The optimum formulation was found to be one that contained 30% Cyclopentasiloxane , 0% PEG-12 Dimethicone, 10% 18-MEA, 29% water, 10% hair strengthening actives, 12.86% emulsifier combination and 8% other oils.

The study also showed that including hair strengthening actives, such as hydrolysed proteins had significant effects in the tensile strength properties of chemically treated African hair.

KEY WORDS: African hair, braids, lubricity, braid detangling force, coefficient of friction, tensile strength.

Related publications by the author

The basic formulation used in this study has been granted a full South African patent, titled: **HAIR TREATMENT COMPOSITION**, country: **South Africa**, applicant(s): **Nelson Mandela Metropolitan University**, inventor(s): Mkentane, Kwezikazi and Vorster, Nicole, ref no: p2122za00/ev/ns.

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CHAPTER 1

INTRODUCTION

A large number of people throughout the world have naturally kinky hair that may be very difficult to manage. These people often subject their hair to vigorous and harsh treatment processes in order to straighten it and hence make it more manageable. Examples of these processes which may cause damage to the hair include chemical relaxing, heat styling, UV and mechanical processes [1]. As a result of these treatments the hair may become fragile and susceptible to damage.

1.1 AFRICAN HAIR PRACTICES

The geometrical structure of African hair makes the hair difficult to manage. African hair has kinks and twists along the hair length; the diameter and cuticle size also differ along the hair shaft. The structure of African hair results in the hair having several fragile points , making hair damage a concern for people of African descent, even for those with chemically unaltered hair (natural or virgin). Several hair practises prevalent in African hair-care sometimes further compromise the health of African hair [2].

1.1.1 Chemically straightened hair practices

Chemically straightened hair is generally refered to as “relaxed” hair. Relaxing African hair straightens the coiled structure irreversibly. This is achieved by treating the hair with formulations containing caustic alkalis through lanthionine formation. Relaxers work by breaking disulfide bonds present in the hair structure as follows[1]:

$$\underset{\text{Cystine}}{\text{K-S-S-K}} + \underset{\text{hydroxide}}{\text{OH}^-} \longrightarrow \underset{\text{lanthionine}}{\text{K-S-K}} + \text{OH}^- + \text{S}^{2-}$$

The frequency of hair-straightening treatments (relaxing) depends on the rate of hair growth since the relaxers are only applied to new hair growth. Relaxing hair can be damaging to hair and scalp because of the irritating nature of relaxing actives. The breaking of the hair’s disulphide bonds through relaxer treatment weakens the hair. Once the hair has been chemically treated, it can either be heat styled, weaved or braided. Damage incurred when mechanical stress is applied to chemically treated hair, such as is the case when the hair is braided or weaved,has been termed as “chemo-mechanically damaged hair’[2].

1.1.2 Natural hair practices

Natural hair is hair that has not been exposed to any chemical treatment. Natural hair is stronger than chemically treated hair. Several options are available for people with natural hair; these are shown in table 1.1[3].

Table 1.1 Hair practises for people with natural hair.

Natural hair practises
Heat Styled
Dreadlocks
Braids
Twists
Afro
Shaved

Hair braiding is a popular and fashionable trend amongst many people, in particular people of African descent. When braided, the hair does not have to be relaxed or heat treated, and may be washed less frequently. Braided hairstyles serve to preserve hair and protect it, and to give it time to rejuvenate after a period of harsh treatment.

During the braiding process synthetic braids are attached to natural hair by weaving a length of the natural hair into one end of each braid. Several strands of natural hair are used to secure each braid. The braids are normally left intact for a number of weeks or even months [2,4].

The process of detangling the braids is known as the “take down” process. This process involves the braids being cut just below where the natural hair ends, and the hair being untangled from the remaining parts of the braids.

The take down process is complicated by the fact that the braids are often cemented to the hair strands due to the build-up of sebum or oil that mixes with dirt. This phenomenon is known as “matting” and results in the braids being securely attached to the hair strands through the formation of “clumps”. These “clumps” are difficult to remove and may result in hair breakage, damage or hair loss.

The take down process is further complicated by the fact that dead hair or hair detached from the scalp may get entangled with other hair causing knots. These knots are difficult to remove and may result in further hair breakage, damage or hair loss. Depending on the tightness and the size of the braids, as well as the degree of matting or knotting, it may take between 4 to 8 hours to remove the braids.

There is, accordingly, a need for a hair treatment composition that will facilitate the take down process, shorten the amount of time spent removing the braids and minimize the amount of damage, hair loss or breakage by improving the strength, elasticity and overall condition of the hair.

1.2 PROJECT BACKGROUND – 2009 STUDY

The development of a formulation designed to facilitate the process of detangling braids from African hair commenced in a 2009 study. The study investigated the factors that influence the ease of braid detangling. The study investigated and compared the attributes of existing braid detangling formulations with the aim of improving them and developing a similar formulation for local consumers. The existing formulations were at the time not readily available to the South African consumer. The study found that braids could be detangled faster by employing the new formulation. The formulation developed has since been granted a South African patent [5].

1.3 BACKGROUND OF CURRENT STUDY

The aim of the current study was to further investigate the factors identified in the 2009 study and to further develop and optimise the 2009 formulation. To address the time and labour issue of taking down braided hairstyles other workers have designed, patented and marketed various braid detangling and braid removal tools [4]. Several braid removal creams are also purchasable online for braid wearers to facilitate in the braid removing process [6, 7, 8, 9].

1.3.1 Existing braid detangling aids

1.3.1.1 Braid detangling tools

To address the labour intensity of the braid detangling process, designers have invented braid detangling or braid removing tools. These tools are listed below in table 1.2 and will be discussed in greater detail in chapter 3.

Table 1.2 Braid detangling tools

Name	Inventor	Year
1. Debraiding tool	Taylor et al	1997
2. Braid detangling device	Carty et al	1997
3. Tool for removal of braids in hair	Robinson et al	2000
4. Braid removal tool	Phillips et al	2000
5. The Unbraider		2010
6. Braid removal tool	Love-Johnson et al	2011

1.3.1.2 Braid detangling formulations

Manufacturers of hair -care products have also worked to address the challenges of braid detangling by developing braid detangling formulations. Listed below are some of the braid detangling formulations available to the international consumer.

1. TAKEDOWN range of products[6]
2. Royal roots detangler[7]
3. Treasured locks knots no more[8]
4. Murray's Unlock quick release braid remover[9]
5. Better Braids Medicated Unbraider[10]

The above braid removal formulations contain high volumes of water, cationic surfactants and silicones. The functional actives of braid detangling formulations are discussed in detail in the chapter 2.

1.3.2 Proposed formulation

The test formulation investigated in this study was derived from a Rasta hairstyle [11] removal formulation and two commercial braid removal creams. The formulation was customised in order to meet the requirements of a successful braid detangling formulation. This can be achieved by making use of ingredients such as surfactants, oleo chemicals, olefins, esters and silicones. The development of the formulation is discussed in greater detail in chapter 2 of this dissertation.

1.4 RESEARCH HYPOTHESES

In this current study, it is hypothesized that:

1. Introducing a lubricating formulation between the hair and synthetic hair that make up the braid, will decrease the friction between the two surfaces, while also transporting foreign particles (matting; sebum mixed with dirt) away from the two surfaces, and will make the overall process of removing braids easier and quicker. The lubricating formulation will “slicken” the braid and hair combination, allowing them to be pulled apart or detangled with ease.

   This hypothesis is broken up into two parts:

   a) Introducing a lubricating formulation to braided hair in order to decrease the braid detangling forces and

   b) Introducing a lubricating formulation to unbraided hair to decrease the frictional forces between human hair and synthetic hair.

2. Including hair strengthening actives like hydrolysed proteins in the formulation will increase the tensile strength of hair and repair damaged hair by binding strongly onto the hair shaft, thereby increasing the threshold point at which hair would normally break and minimize hair damage and subsequent hair loss.

1.4.1 Response variables of current study

The response variables that were chosen as indicators of the efficacy of the braid detangling formulation are;

1. The ease of braid detangling; this was measured using a customized comb test to measure the force required to detangle the braid.
2. The friction of the hair that would be within the braid; friction measurements were carried out using an industrial tensometer.

3. The tensile strength of the hair treated with the test formulation containing hair strengthening actives in order to minimize hair damage. Tensile testing was conducted using a tensometer.

1.5 STRUCTURE OF STUDY AND DISSERTATION

The research hypotheses proposed above are discussed in the following chapters of this dissertation. Chapter 2 discuss the product development of the test formulation used. Chapter 3 is the first experimental chapter and it discusses the first part (a) of the first hypothesis mentioned above, using ease of braid detangling as a measure of determining the extent of lubrication. Chapter 4 is the second experimental chapter and it addresses the second part (b) of the first hypothesis, investigating the friction forces between human hair and synthetic hair. An experimental design is employed in chapter 4 in order to evaluate the effects of varying the levels of actives in the formulation. Using the results of the experimental design, the formulation was then optimised. Chapter 5 is the third and last experimental chapter which investigates the second hypothesis of theoretically minimizing hair damage by increasing hair tensile strength. Chapter 6 summarises the work and discusses the conclusions reached in the study.

1.6 REFERENCES:

1. Holloway V. Ethnic cosmetic products. Dermatol Clin 2003; 21:743-749
2. Nonhlanhla. P Khumalo, Susan Jessop, Freedom Gumedze, Rodney Ehrlich. Determinants of marginal traction alopecia in African girls and women. J Am Dermatol 2008;59:438-9
3. Ingrid E. Roseborough, Amy J. McMichael. Hair care practises in African American patients. Sem Cut Med Surg 2009;23:103-108
4. Braid detangling tools. See references 1 -6 in chapter 3
5. South African patent application: 2010/0428~ZA. Hair treatment composition.
6. www.takedownproducts.com (last accessed: November 2011)
7. http://www.treasuredlocks.com/howtorelo.html (last accessed: February 2011)
8. http://www.treasurelocks.com/desore.html#item.Tabs(last accessed: February 2011)
9. http://item.mobileweb.ebay.com/viewitem?itemid=370565422078&cmd=VIDESC
10. www.keystone-labs. (last accessed January 2012)

11. Maillefer et al, Kalbfleish, Rehmann and Chambettaz. Methods of setting up, caring for and later removing a temporary Rasta hairstyle. United States patent application # 20040028632.

CHAPTER 2

THE FORMULATION OF A BRAID DETANGLING COMPOSITION

2.1 PRODUCT DEVELOPMENT

The road to product development often stems from a problem that needs to be solved or addressing an opportunity that has been identified. The product development process is outlined below [1]:

Idea generation → idea screening → concept development and testing → business analysis

The current study started in much the same way outlined above. The idea of a braid detangling product started in 2009 with the identification of a need in the local ethnic hair care market for a cosmetic formulation that facilitates the process of detangling braids. The current study is on the third step of the product development process outlined above.

2.1.1 Problem identified

The manual process of taking down braids without any aid is time consuming [2-6] and a lot of hair loss occurs as a result of oil build-up, causing hair and synthetic fibres to bind together. Even once the braids are removed, considerable effort is still required to take down braids and to detangle hair once braids are removed. This research looked at the factors that affect the process of taking down braids. A challenge that had to be overcome was the lack of published literature on the subject and difficulties of the braiding process and removal of braids. As a result of the lack of published research on the topic, the researcher relied heavily on her own observations and on the opinions of fellow braid wearers. The factors in table 2.1 were observed to be amongst the key factors that influence the ease with which manufactured hair is detangled from a braid wearer's natural hair.

Table 2.1 Factors that influence the ease of braid detangling

1. Braid size	Thicker braid = easier removal, less time
2. Force exerted during braiding process	Tighter braid = harder to detangle
3. Texture/ condition of hair	Silkier hair = easier braid removal
4. Length of hair within braid	Longer = harder or more time consuming
5. Type of manufactured hair used	Silkier hair = easier detangling
6. Hair moisture content	Dry = harder, caution must be exercised because hair more brittle
7. Dandruff & oil build-up (matting)	Matted braid = harder (newer/fresher braids are easier to remove than older braids as far as matting is concerned)
8. Braided region	Hair line is hardest to detangle. Caution must also be exercised in this region because care must be taken since hair is weakest in this region [2].

Most of the factors that were observed are factors that can only be controlled by the stylist during the braiding process. The purpose of this study was to investigate only the factors that could be influenced by the braid wearer when the time came to remove their braids. In order to investigate these factors, a cosmetic formulation was proposed. The attributes of a “hypothetically successful” formulation for a braid detangling can be summarised in the next section:

2.1.2 Attributes and objectives of a braid detangling formulation

The attributes and objectives of a braid detangling formulation are the following:

- ✓ To soften and detangle hair, thereby allowing hair to be loosened from braids with minimal effort.
- ✓ To increase the lubricity of the hair within the braid, thereby allowing the hair to “slip” out of the braid.

- ✓ To increase the tensile strength of hair, in order to minimize hair breakage while hair is being detangled from the braid.
- ✓ Be safe and easy to use: the ingredients used in the proposed formulation have to adhere to industry regulations. The Cosmetic Ingredient Review (CIR) panel was used as the acceptable standard in this study. The CIR is an independent review programme for cosmetic ingredients. The CIR has participation from the U.S Food and Drug Administration (FDA).
- ✓ To dissolve oil build- up of dead hair that has been mixed with dandruff flakes, oil and dirt from the environment.

The desired attributes of a successful braid detangling formulation are further analysed in table 2.2 in order to investigate what combination of active ingredients could address the problems associated with the removal of braids.

Table 2.2 Desired attributes of a braid detangling formulation

Action	Influence / result	How? (actives ingredient)	Test
1. Lubricate and detangle	Helps loosen braids from hair	Lubricant = silicone derivatives	Friction measurements Treated hair vs. untreated hair
2. Dissolve matting	No matting = less breakage	Good cationic surfactants to solubilize hair matt	
3. Strengthen hair	Stronger hair = less hair damage	Hydrolysed protein actives	Tensile strength measurements
4. Moisturise	Moist hair, less brittle Less breakage	Humectants	Tensile strength measurements

2.2 BASE FORMULATION -2009 STUDY

As a starting point for formulating a braid detangling formulation, a formulation used to remove a Rasta hairstyle was used alongside two braid detangling formulations. The composition to remove a Rasta hairstyle is disclosed in U. S patent application no. 20040028632 [7].The patent discloses methods of setting up, caring for and later removing a temporary Rasta hairstyle. Setting up of the Rasta hairstyle, also known as “dreadlocks” entails the application of a roughing substance that increases the adherence of hair fibres in order to allow them to interlock. The roughening is achieved by back combing the hair and using silica as a roughening substance. To remove the temporary Rasta hairstyle a composition that increases the combability of the hair is prescribed. The two-phase composition (composition C) is said to increase the combability of human hair and the dreadlocks are cautiously combed out from the hair tips to the hair roots with a hand comb. Table 2.3 shows composition C as described in the patent application.

Table 2.3 Rasta Hairstyle Removal Composition

Phase A (oil phase)	**% present in formulation**
Cetearyl alcohol	6
Vaseline®	1.35
Paraffinum Perliquidum (Mineral oil)	1.2
Cetyl trimethyl ammonium chloride	1
Silicone oil	0.5
Phase B(aqueous phase)	
Lanolin alcohol	0.3
Lanolin	0.15
Citric acid	0.5
water	to 100

The Rasta removing composition was used as part of the base formulation because it was said to improve the combability of hair that had been temporarily molded into a dreadlock hairstyle. The two braid removers that were also included in the base formulation were the "Royal roots detangler, softener and braid remover" [8] and the "Knots no more cream" [9]. Table 2.4 shows the ingredients present in these two braid detangling formulations as listed on the containers.

Some common attributes were found in the compositions of the two commercial braid detanglers and the Rasta removal formulations; all three formulations contained cationic surfactants, fatty alcohols, moisturizers, vegetable or silicone oils and water. Although no published scientific literature could be found for the two braid detanglers in table 2.4, they are marketed in the United States as efficient braid removers [8, 9].

Table 2.4 Braid detangling formulations

Royal Roots Remover	Knots No more Cream
Water	Water
Nut Oil	Hybrid Safflower Seed Oil
Propylene	Propylene Glycol
Vitamin E	Cetyl Alcohol
Olive Oil	Dicetyldimonium Chloride
Cocoa Butter Oil	Benzalkonium Chloride
Herbal Moisturizers	Hydroxy ethyl cellulose
Detanglers	Organic Guar Gum
Silk Protein	Glyceryl Stearate
Balsam	Fragrance
Aloe Vera	Sorbic Acid

The “Royal roots detangler, softener and remover” is described by its marketers [8] to be a highly versatile product that:

- Removes braids, weaves and dreadlocks
- Can be used as a conditioner
- Can be used as a detangler, to soften and to reduce frizz

“Treasured Locks Knot No More^{TM}” is a leave-in detangling conditioner that is reported to be especially formulated to facilitate the removal of hair styles such as locks, twists, cornrows and braids. The product is marketed as a product that is specially made to soften and "slicken" the hair making it possible to remove knots and tangles that would otherwise cause pain and breakage in the braid detangling process [9]. The “Treasured Locks Knot No More^{TM}” cream is said to:

- Reduce the friction between the hair strands, allowing one to pull a comb or their fingers through the hair to remove braids or tangles with minimal hair loss and effort.
- Improve the hair's elasticity, minimizing breakage while pulling through the tangles.

The functional active ingredients of the commercial braid detanglers matched the required attributes of an efficient braid detangler. The attributes of an efficient braid detangler were discussed earlier in table 2.2. Active ingredients with the same functionalities as those seen for the three formulations discussed above were used as a guideline for the formulation of a base formulation. Table 2.5 below shows the active ingredients chosen for the base formulation in the 2009 study and their respective roles

Table 2.5 Base formulation (2009 study)

	Functional Ingredients	Function in hair care
1.	Cyclopentasiloxane	Conditions, lubricates and imparts softness and silky feel
2.	Dimethicone	Detangles and improves shine
3.	Isopropyl palmitate	Imparts silkiness, lubricity, synthetic moisturizers
4.	Cetyl Alcohol	Humectant & emulsifier, smoothens & softens hair cuticle
5.	Cetyl trimethyl ammonium chloride	Cationic surfactant
6.	Hydroxyethyl cellulose	Binder, thickener and stabilizer
7.	HYDROLASTAN	Protective hydrolysed protein, strengthener
8.	SETAKOL®	Hydrolysed silk protein
9.	Lanolin	Moisturizer
10.	Sodium Oleate	Anionic surfactant
11.	Olive Oil	Essential oil that also serves as a lubricant
12.	Tea Tree Oil	Lubricant and fragrance

The finding of the 2009 study was that braids could be detangled more easily when using a combability increasing formulation to facilitate the process. It was observed that the formulation gave rise to a soft-feel perception of the hair. The response variable for the study was the amount of time taken to remove the braids from a wearer's head [10] .Only one subject was used for the study. The results for the 2009 study can be found in appendix 2.1.

From the literature review of existing braid detanglers, basic hair conditioning formulations were seen to both match the key ingredients found in braid detanglers and to meet some of the criteria for the desired attributes of a new efficient braid detangler. The key ingredients used in hair conditioners are listed in table 2.6. Hair conditioning formulations are said to cause changes in the hair's surface properties [11]. The efficiency of hair conditioners can be measured quantitatively using coefficient of friction measurements and qualitatively by using human perception of feel. Hair conditioners work by thinly coating the hair strands via Van der Waals forces of attractions [11, 12]. This observation relating to hair conditioners was used as a guideline in choosing response variables that could be measured in evaluating the efficacy of a braid detangling formulation. The primary function of a hair conditioning formulation or product is to make hair easier to comb [11].To measure the efficiency of hair conditioners, friction and combability measurements are generally chosen to be reliable test parameters. Hair combability is dependent on the lubrication of the fibre surface. The fibre surface can be lubricated by the sorption or binding of lubricating or conditioning ingredients to the hair fibre surface [11].

Table 2.6 Hair conditioner ingredients and their benefits[11]

Conditioner actives for benefits in wet environment

Key ingredient	Benefits
Cationic surfactant	Creamy texture
Fatty alcohols	Ease of spreading
Water	Slippery feel while applying and soft rinsing feel

Conditioner actives for benefits in dry environment

Key ingredient	Benefits
Silicones	Moistness
Fatty alcohols	Softness
Cationic surfactant	Dry-combing ease

Tables 2.7 through to 2.9 show examples of different types of hair conditioning formulations [11].

Table 2.7 Example of a simple hair conditioner

Ingredient	% in formulation
Cetyl trimethyl ammonium chloride	1.0
Cetyl alcohol	2.5
Hydroxy ethyl cellulose	0.5
Fragrance	0.2
Preservative (Germaben II)	0.5
Citric acid	0.2
Water	q.s to 100%

Table 2.8 Example of a complex hair conditioner

Ingredient	% in formulation
Cetyl alcohol	1
Stearyl alcohol	1
Hydrolysed animal protein	<1
Stearamidopropyl dimethyl amine	<1
Cetearyl alcohol	<1
Propylene glycol	<1
Keratin polypeptides	<1
Aloe	<1
Tocopherol	<1
Panthenol	<1
Preservative	<1
Fragrance	<1
Water	q.s to 100%

Table 2.9 Example of a "deep" hair conditioner

Ingredient	% in formulation
Phase A (oil phase)	
Cetyl alcohol	6.0
Stearamidopropyl dimethyl amine	1.5
Mineral oil	0.5
Propylene glycol	1.0
Phase B (aqueous phase)	
Citric acid	0.2
dicetyl dimethyl ammonium chloride	1.0
Germabem II	0.5
Fragrance	0.4
Water	q.s to 100%

2.3 BASE FORMULATION –CURRENT STUDY

The formulation in this current study is an improvement of the 2009 formulation. In the current study four components were varied using a d-optimal mixture design in order to study their effects on the lubricity and tensile strength of human hair. The main actives chosen and varied in this current study are: Cyclopentasiloxane, PEG-

12 Dimethicone, 18-Methyl Eicasenoic Acid (18-MEA) and water. The silicone derivatives Cyclopentasiloxane and PEG-12 Dimethicone were used because of their lubricating ability. These were hypothesized to decrease hair friction and 'slicken' the braided hair in order to allow the hair and the synthetic fiber to be pulled apart with ease.18-MEA replaced cetyl alcohol in table 2.5 and was included in the test formulation because of its reported ability to replenish the hair fibre [13]. Damage to the hair cuticle increases hair friction, making the hair susceptible to tangle and subsequent breakage [11-13]. The water was the solvent for the water-soluble phase (phase B). The following sections discuss the active ingredients of the base formulation used for the current study.

2.3.1 Cyclopentasiloxane (D5)

Silicones are used as effective lubricating actives because when in a formulation, the silicone molecules remain as droplets surrounded by water. The silicone molecule's high molecular weight causes the molecules to remain liquid and to drain off the hair surface gradually. This gradual draining process is said to create a long-lasting, soft and smooth feel for hair that has been treated with a product containing lubricating silicone actives [12]. Cyclopentasiloxane has good spreading and lubrication which makes it an ideal carrier for other ingredients and an good active for light hair conditioning formulations [14]. Figure 2.1 shows the chemical structure of this silicone.

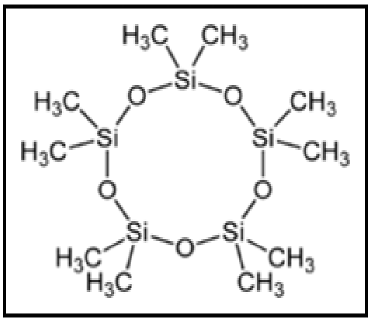


Figure 2.1 Chemical structure of Cyclopentasiloxane

The characteristics of Cyclopentasiloxane are listed below [14]:

- ✓ Emollient
- ✓ Insoluble in water
- ✓ Non-sticky applications

An emollient is an ingredient that has a softening or smoothing effect. The benefits of including Cyclopentasiloxane in hair products are that it:

- ✓ makes hair easier to brush
- ✓ Adds silky softness and shine to hair formulations
- ✓ Improves spreadability and provides lubricity to hair formulations
- ✓ Has volatility in end use applications, therefore, no build-up.

2.3.2 PEG-12 Dimethicone

Dimethicone is used in hair conditioners and shampoos to improve hair separation and shine, to protect the hair and to add sheen and softness [15]. Figure 2.2 shows the chemical structure of dimethicone.

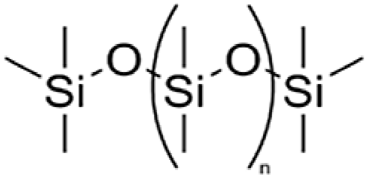


Figure 2.2 Chemical structure of PEG-12 Dimethicone

Characteristics of PEG-12 Dimethicone [15]:

- ✓ silicone based polymer
- ✓ optically clear
- ✓ Safety: Inert, non-toxic and non-flammable

Benefits of dimethicone in hair care:

- ✓ Adds lustre and sheen
- ✓ makes hair shiny and slippery

2.3.3 18-Methyl Eicasenoic Acid (18-MEA)

The human hair fibre contains natural 18-Methyl Eicasenoic Acid (18-MEA) on its hair surface; however the 18-MEA is lost through environmental and mechanical stresses. 18-MEA is described by Steven et al [13] to be an unusual branched-chain fatty acid that binds covalently onto the cuticle surface of the hair shaft. The presence of this fatty acid creates a lubricating monolayer on the hair shaft. Changes arising from the presence of 18-MEA on the structure of the hair shaft can be studied using atomic force microscopy. Figure 2.3 shows the binding of 18-MEA onto the hair shaft.

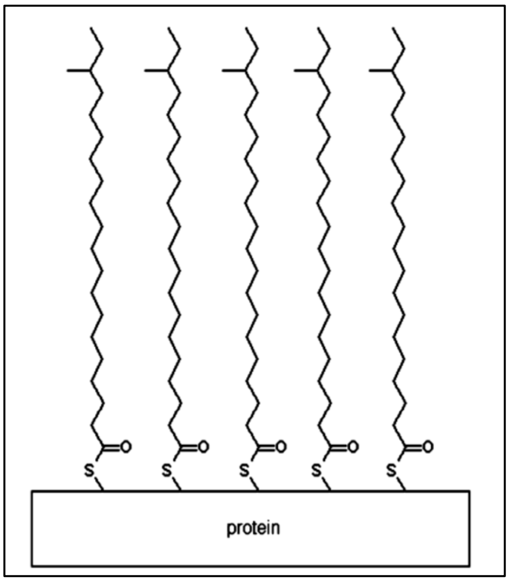


Figure 2.3 binding of 18-MEA onto the outer layer of hair shaft.

The 18-MEA is covalently attached to the cysteine-containing proteinaceous outer hair surface of a hair via a thioester linkage. The characteristics and benefits [16] of 18- MEA in hair care are that it:

- ✓ Replenishes lipid surface of hair
- ✓ Makes hair more hydrophobic
- ✓ Improves wet and dry combing
- ✓ Restores integrity of hair surface
- ✓ Makes hair less susceptible to environmental stress

- ✓ Adds lubricity and shine
- ✓ Safety: Colourless and odourless and non-stinging

As mentioned earlier, the active ingredients discussed above were varied in formulations in accordance to a d-optimal mixture design. The next section, from paragraph 2.3.4 discusses the ingredients present in the base formulation that were not varied but kept constant in all the formulations that were prepared. A total of twenty (20) formulations were prepared, with different levels of the Cyclopentasiloxane (D5), PEG-12 Dimethicone, 18-MEA and water. An example of one of the test formulations prepared is given at the end of this section. The full experimental design is can be found at the end of this chapter in table 2.12.

2.3.4 Isopropyl Palmitate (IPP)

Isopropyl palmitate is an ester of palmitic acid from coconut oil and is used in hair styling aids and sheen sprays [17]. IPP acts as moisturizer and antistatic agent in hair care formulations. It is a colourless, almost odourless liquid that is said to impart silkiness to skin and hair [17]. The chemical structure of IPP is shown in figure 2.4.

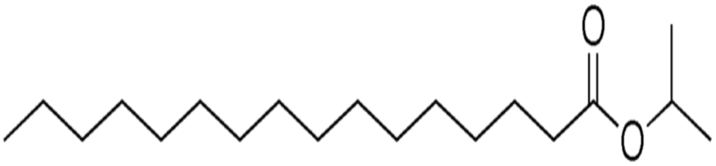


Figure 2.4 Chemical structure of Isopropyl palmitate

2.3.5 Cetyl trimethyl ammonium chloride (CTAC)

Cetyl trimethyl ammonium chloride is a functional cationic surfactant that is used in hair conditioning products. It neutralizes electrostatic charges on the hair surface by attaching onto the hair’s negatively charged surface [11]. Figure 2.5 below shows the chemical structure of CTAC.

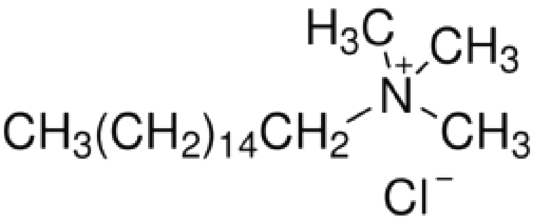


Figure 2.5 Chemical structure of cetyl trimethyl ammonium chloride

2.3.6 Hydroxyethyl cellulose (HEC)

Hydroxyethyl cellulose, abbreviated to HEC, is a thickener that is derived from cellulose. It is widely found in cosmetic creams and lotions. The chemical structure of HEC is shown in figure 2.6.

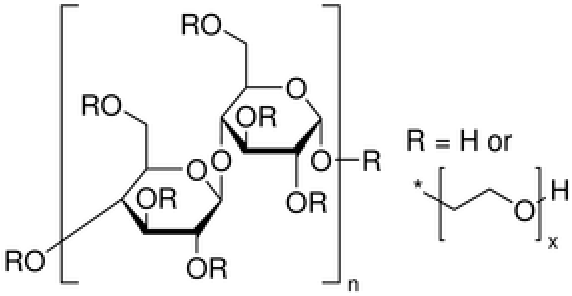


Figure 2.6 Chemical structure of HEC

2.3.7 Hydrolysed Elastin (HYDROLASTAN, available from Pentapharm [18])

Hydrolysed Elastin is derived from elastin, a flexible protein extracted from the skin of plants or animals. HYDROLASTAN is a non-viscous, amber coloured, clear to slightly opalescent solution which is clearly fluorescent under UV-light. It is obtained from bovine ligaments by enzymatic degradation. HYDROLASTAN is regarded as a protective protein with affinity to skin and hair. This ingredient is said to be suitable for skin care and hair care formulatory because it has a high substantivity for both skin and hair. HYDROLASTAN also provides smoothing properties to skin and hair. The suggested levels of use for HYDROLASTAN are 2 – 5% [18].

2.3.8 Hydrolysed Sericin (SETAKOL®)

SETAKOL® is a hydrolysed silk protein that is manufactured and marketed by Croda, a manufacturer of chemicals for the cosmetic industry. SETAKOL® is said to have a unique affinity for proteins. This affinity for proteins allows SETAKOL® to strongly bind to the keratin of the skin and hair, forming a biological, multifunctional protective film. This ingredient is said to get right inside the hair cuticle, to cement and seal the outer scale layer and forms an adhesive, biological, protective film [19]. With suggested levels of use of 2-5 %, SETAKOL® is also said to strengthen the hair fibre and to give a conditioning effect to stressed hair [19]. The INCI name for SETAKOL® is hydrolysed Sericin.

2.3.9 Lanolin

Lanolin is a moisturising agent that is found in sheep's wool. Lanolin is used in the cosmetic industry because of its unique composition of complex sterols, fatty alcohols, and fatty acids.

2.3.10 Olive oil

Olive oil is an essential oil that functions as a lubricant and carrier oil in hair care products.

2.3.11 Oleth-5

Oleth-5 is an ethoxylated oleyl alcohol that is used in personal care as a non-ionic emulsifier. Oleth-5 was selected as a suitable emulsifier to aid the mixing of the oil phase with the water phase. An emulsifier is a surface active agent that promotes the formation and stabilization of an emulsion by reducing the surface tension of water. The surface activity of an emulsifier is related to its hydrophile-lipophile balance (HLB). HLB is determined by the size of the hydrophilic (water-loving or polar) portion of the molecule as compared to the size of the lipophilic (oil-loving or non-polar) portion of the molecule. A cosmetic emulsion can be stabilized by using emulsifiers that match up with the ingredients in the formula. Ingredients with low required HLB values need low HLB emulsifiers. Ingredients with high required HLB values need high HLB emulsifiers.

The HLB of oleth-5 is 8.8. This HLB value matched the calculated required HLB for the mixture. Other emulsifiers present in the formulation were cetyl trimethyl ammonium chloride and lanolin.

2.4 PREPARATION OF FORMULATION

Table 2.10 below shows a typical test formulation that was used in the study. In total, 20 test formulations were prepared; these are shown in the full experimental design in table 2.12.

Table 2.10 Example of a typical test formulation used in the current study

Ingredient	**Test Formulation 1**
Phase A (oil phase)	**% present in formulation**
Cyclopentasiloxane	0
PEG-12 Dimethicone	5.0
18-Methyl Eicasenoic acid (18-MEA)	5.0
Isopropyl Palmitate (IPP)	3.0
Oleth -5	10.0
Olive oil	5
Phase B (aqueous phase)	
Hydroxy ethyl cellulose	0.2
SETAKOL®	5.0
HYDROLASTAN	5.0
Cetyl trimethyl ammonium chloride	2.5
Lanolin	0.15
Distilled water	Qs to 100

2.4.1 Manufacturing procedure:

The test formulations were prepared in accordance to the preparation procedure for oil-in-water emulsions [11, 20]. The following procedure was used:

1. In a 250 ml beaker, dissolve all the water soluble ingredients (phase B) into q.s amount of distilled water.
2. Place mixture onto a hot place and raise heat of mixture to 60^0C. Stir mixture continuously to dissolve all solids.
3. In a second 250 ml beaker, heat the oil-soluble components to melt all the solids. Do not exceed 70^0C for heating. This is phase A.

4. With the aid of an IKA T18 Basic ®, Ultra Turrax homogeniser, set on the second speed setting, mix the two phases by slowly adding phase B to A with continuous stirring.
5. Continue stirring for 10-15 minutes to form a rich creamy emulsion, and then cool to ambient.

The stirring speed and type of mixing aid, rate of cooling and order of phase addition all influence the consistency of the formulation prepared [11]. Figure 2.7 shows a schematic diagram of the manufacturing set up.

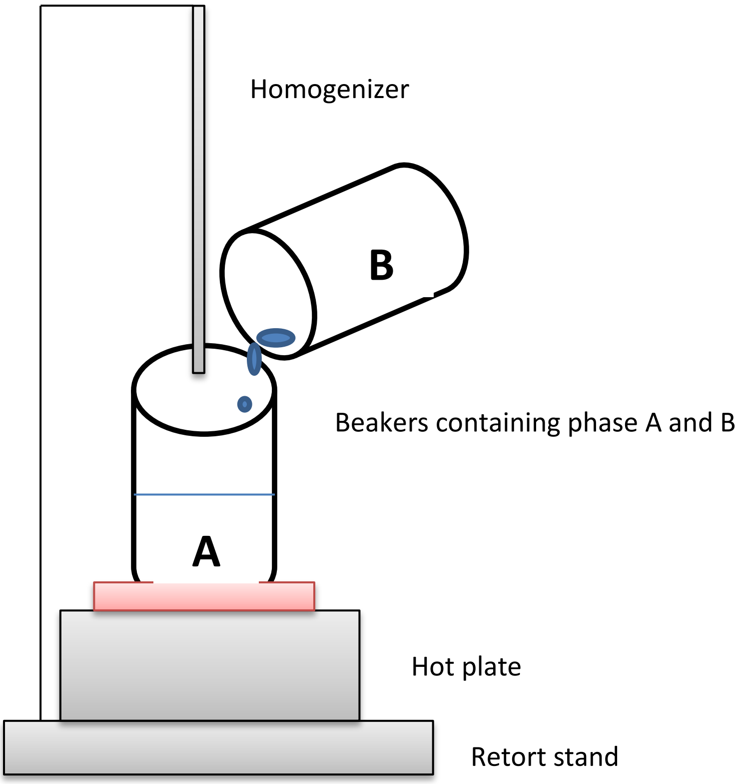


Figure 2.7 Schematic diagram of the manufacturing set up.

The ingredients discussed in section 2.3 were identified to be theoretically suitable to affect the desired results in an efficient braid detangling formulation. The desired attributes of an efficient brad detangling formulation were listed in table 2.2. All the selected ingredients have been reviewed and approved for use in cosmetic products by the Cosmetic Ingredient Review (CIR). Table 2.11 shows the details of each of the ingredients used.

Table 2.11 Table of materials

Ingredient	**Supplier**	**Grade/Purity**
Cyclopentasiloxane	Ethnichem	Cosmetic
PEG-12 Dimethicone	Dow Corning	Laboratory
18-Methyl Eicasenoic Acid	Croda SA	Cosmetic
Isopropyl Palmitate	Sigma Aldrich	>90%
Oleth-5	Ethnichem	Cosmetic
Olive oil	Aoeliko	
Hydroxy ethyl cellulose	Sigma Aldrich	Technical
SETAKOL®	Pentapharm	Cosmetic
HYDROLASTAN	Pentapharm	Cosmetic
Cetyl trimethyl ammonium chloride 25% wt. % in H_2O	Sigma Aldrich	Technical
Lanolin	Sigma Aldrich	Technical
Distilled water	NMMU	Laboratory

2.5 FULL EXPERIMENTAL DESIGN

Table 2.12 shows the full experimental design that was used in the study. In chapters 3 and 5, only selected test formulations are used, whilst in chapter 4, the full experimental design is used. The ingredient levels were selected according to current and suggested levels of use for each ingredient.

Table 2.12 Full Experimental design

(n)	Cyclopentasiloxane (D5)	PEG-12 Dimethicone	18-MEA	IPP	Olive Oil	Oleth-5	SETAKOL	HYDROLASTAN	HEC	CATC	Lanolin	Water
1	0.00	5.00	5.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	59.00
2	0.00	10.00	10.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	49.00
3	5.63	2.50	7.50	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	53.38
4	5.00	5.00	0.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	59.00
5	7.50	10.00	5.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	46.50
6	20.63	2.50	2.50	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	43.38
7	30.00	0.00	0.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	39.00
8	10.00	0.00	0.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	59.00
9	15.00	10.00	5.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	39.00
10	0.00	0.00	10.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	59.00
11	15.00	5.00	0.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	49.00
12	0.00	10.00	0.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	59.00
13	10.00	0.00	0.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	59.00
14	20.00	0.00	10.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	39.00
15	15.00	0.00	5.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	49.00
16	0.00	10.00	0.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	59.00
17	0.00	0.00	10.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	59.00
18	20.00	0.00	10.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	39.00
19	0.00	10.00	10.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	49.00
20	25.00	5.00	0.00	3.00	5.00	10.00	5.00	5.00	0.21	2.50	0.15	39.00

REFERENCES:

1. http://en.wikipedia.org/wiki/New_product_development last accessed: 13th January 2012

2. Taylor et al, inventor; Joseph Taylor, Brenda M Taylor. Debraiding tool. United States Patent 5071920. 1997 Dec 30.

3. Carty et al, inventor; Jennifer Carty, Kenneth Carty. Braid Detangling Device. United States Patent 5911225. 1997 Jun 15.

4. Robinson, inventor; Angela Mae Robinson. Tool for the removal of braids in hair. United States Patent 6095154. 2000 Aug 1.

5. Love-Johnson, inventor; Deborah Love-Johnson, Shawnee. Braid removal tool, United States Design Patent D6361225. 2011 April12.

6. Phillips, inventor; Alton Phillips. Braid removal tool. United States Patent 602 1783.2000 Feb.

7. Maillefer et al, Kalbfleish ,Rehmann and Chambettaz, Methods of setting up, caring for and later removing a temporary Rasta hairstyle.
   United States patent application # 20040028632.

8. http://www.treasuredlocks.com/howtorelo.html last accessed: February 2011

9. http://www.treasurelocks.com/desore.html#item.Tabs last accessed: February 2011
10. The development of a braid remover. K. Mkentane, Nelson Mandela Metropolitan University. December 2009.

11. Clarence R. Robbins. The physical and mechanical behaviour of human hair. 3rd Edition. Springer-Verlag

12. Bharat Bhushan. Nanoscale characterization of human hair and hair conditioners. Progress in Materials Sciences, 2008; 53: 585 -710.

13. Steven Breakspear, James R Smith, Gustavo Luengo. Effects of covalently linked fatty acid 18 MEA on the nanotribology of the hair's outermost surface. Journal of structural biology 2005; 149: 235 – 242.
14. www.ewg.org/skindeep/ingredient.php?ingred06=701741
15. http://cosmeticsinfo.org/ingredient_details.php?ingredient_id=811
16. http://www.croda.com/home.aspx?view=dtl&d=content&s=157&r=401&p=2578&prodID=354
17. http://cosmeticsinfo.org/ingredient_details.php?ingredient_id=175
18. www.pentapharm.com/5903say last accessed: November 2011
19. www.pentapharm.com/5208vo lasted accessed: November 2011
20. Juan Carlos, Alva Nieto, Ken-tye Tong. Hair Conditioners: Colloids CE 457/ 527

CHAPTER 3

MEASUREMENTS OF BRAID DETANGLING FORCES

3.1 INTRODUCTION

3.1.1 Hair braiding and braid removal

Hair braiding is a popular and fashionable trend amongst many people, in particular people of African descent. Braided hairstyles serve to preserve and protect the hair, giving it time to rejuvenate after a period of harsh treatment, especially for chemically treated (relaxed) hair. Braided hairstyles are also common amongst people with short hair because any desired length can be achieved. It takes several hours to assemble some braided hairstyles and they are then worn for 4-8 weeks. Braided hairstyles are achieved by interweaving manufactured strands of hair onto the wearer's natural hair in an alternating fashion. For micro or single braids, colloquially known as "singles", the human hair and manufactured strands are braided into 500-1000 micro-thin braids [1].The multiplicity of long micro-thin braids creates the desired effect of long luxurious hair, however there are problems associated with their removal [1-5]. Currently, braid removal is achieved by using fine-toothed combs [3] or safety pins. For three-strand braids, the pointed end of the comb, safety pin or similar tool is put into the braid where the three strands overlap and a force is exerted to loosen and unravel the braid [3]. Figure 3.1 below shows sketches of conventional braid detangling methods. These methods tend to be time-consuming and strenuous, [1] [2] sometimes taking up to a full day or more to achieve for a head of 500-1000 micro-thin braids. These conventional methods also often lead to hair damage [3] as the hair shaft is repeatedly assaulted with the combs or safety pins.

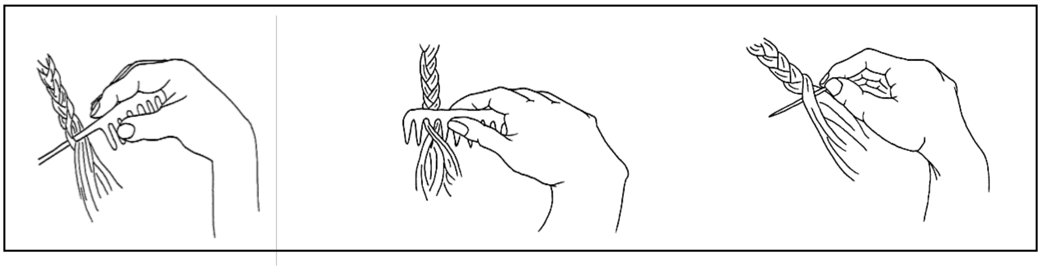


Figure 3.1 Conventional braid detangling methods [3].

The take down process is sometimes complicated by the fact that the human hair within the braids is often cemented together by a mixture of sebum/oil and dirt build-up since efficient hair cleansing while braids are worn is difficult. This phenomenon is known as “matting” and results in the braids being securely attached to the hair strands through the formation of “clumps”. These “clumps” are difficult to remove and may result in hair damage, breakage and/ hair loss.

The take down process is further complicated by the presence of dead hair or hair that has been detached from the scalp that may get entangled with the wearer’s hair that is still intact, causing knots. These knots are difficult to remove and may result in further hair breakage, damage or hair loss.

3.1.2 Braid removal tools

In attempts to resolve some of the problems associated with the detangling of braids, several tools have been designed: US Patent 5701920 to Taylor et a l[1], US Patent 5911225 to Carty et al [2], US Patent 6095154to Robinson et al[3] and US Patent 6361225 to Johnson et al[4], to name a few, all disclose braid removing or braid detangling tools. The latest braid detangling invention is the braid removal tool, US Pat. D636122 to Deborah Love-Johnson [4], as shown in figure 3.2 below. Figure 3.3 shows a braid detangling tool invented by Taylor [1] and figure 3.4 shows the braid detangling device invented by Carty [2]. Carty et al describes a braid detangling device, that when used, would allow the braid wearer to detangle their braids in a shorter time than if braids were removed manually[2] .The use of these and similar inventions to facilitate the braid detangling problem is hypothesized by the inventors to shorten the braid detangling times and effort. [1-5]. US Patent 6095154 to Robinson discloses a braid removing tool with curvature. The curvature of the tool, as shown in figure 3.5, is said to retain the hair to prevent it from slipping out of the tool [3].

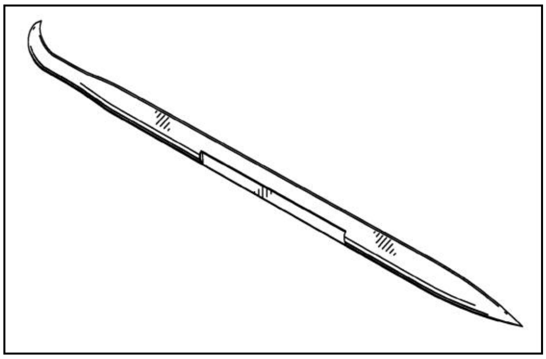


Figure 3.2 The latest braid removal tool (April 2011) [4].

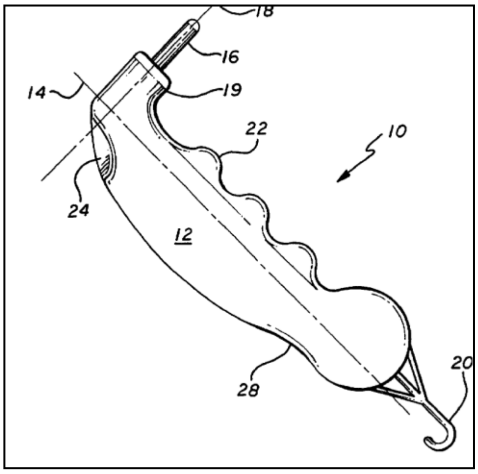


Figure 3.3 Braid removing tool by Taylor [1].

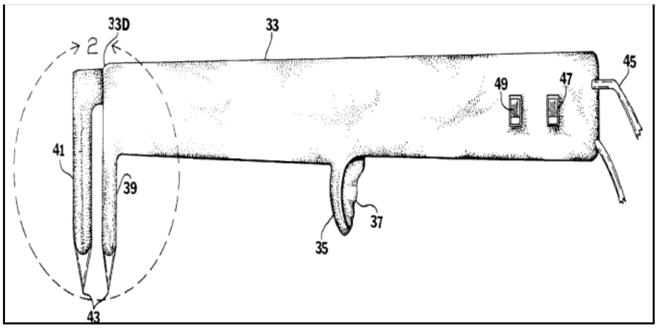


Figure 3.4 A Braid detangling device as shown by Carty in US Patent 5911225[2]

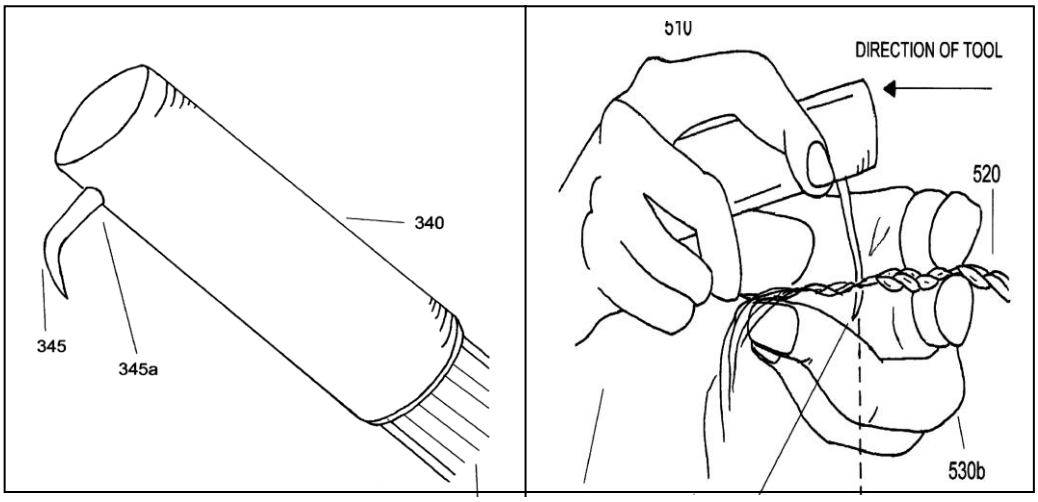


Figure 3.5 A braid removal tool with a curved probe to prevent the probe from slipping out of the braid. [3]

In US Patent 6021783[5] Phillips discloses a braid removal tool that has a comb on a strap and a plurality of prongs. The tool works to remove braids one section at a time when the user engages the tool's prongs with the braids. Using a downward motion, the braids can be combed out. See figure 3.6 .According to Phillips, the tool can be used to remove multiple braids simultaneously [5], effectively shortening the amount of time taken to remove the braids.

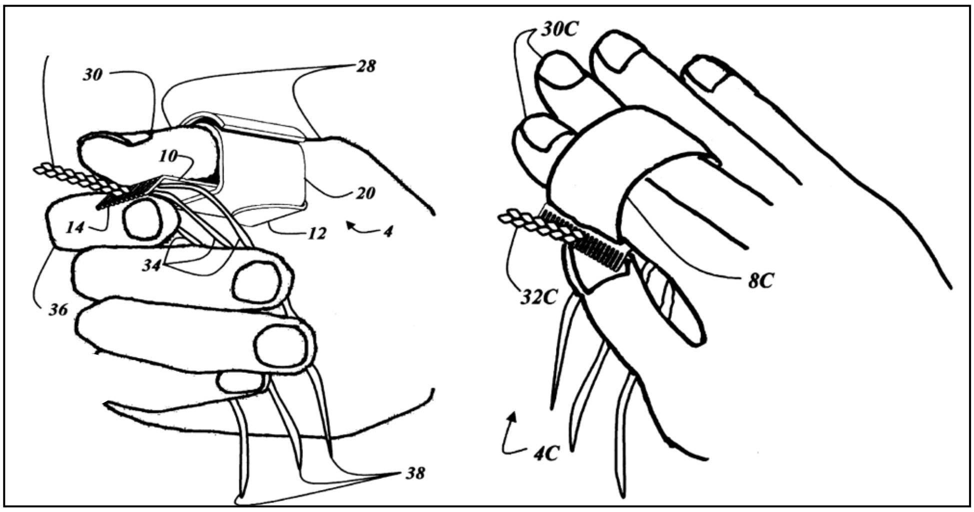


Figure 3.6 A braid removal tool that can remove multi braids simultaneously and can be utilized with one hand [5].

The need to make a braid removal tool commercially available was also identified by a group of women at Glamour Devine [6].The Unbraider is described to be a specialty tool that forms part of a system that makes braid removal easier. This tool is then used in conjunction with a hair conditioner. The Unbraider resembles a wide-toothed comb on one side and a spiky brush on the other (see figure 3.7). The Unbraider is reported to help remove braids with ease and in less time than conventional methods. This tool designed by Glamour Devine is said to remove 6 – 8 braids at a time [6].

Figure 3.7 The Unbraider is said to be engineered to remove 6-8 braids at a time and is used alongside a conditioner [6].

3.1.3 Braid removal formulations

Besides the tools that have been invented to address the challenges faced by braid wearers, braid removing formulations are available to US and UK consumers. These formulations were discussed in detail in chapter 2. Braid removal formulations are said to work by coating the hair shaft with blends of lubricants and water, moisturising and conditioning the hair. None of these existing braid removal formulations is available in South Africa.

3.1.4 Aim of study

The aim of this study was to investigate the effects of formulations similar to those described in chapter 2 in facilitating the braid detangling or braid removal process. The first of these investigations started in 2009 as explained in chapter 2 of this dissertation. The 2009 study on the actives needed to formulate a successful braid detangler resulted in the successful filing of a South African Patent ZA~ 31 2010/04284[7].This patent discloses a hair treatment composition that has the objective of facilitating the take down process in order to shorten the amount of time spent removing the braids. The hair treatment composition disclosed aims to minimize the amount of damage, hair loss or breakage. This was hypothesized to be achievable by reducing the frictional forces within the braid ensemble and by improving the strength, elasticity and overall condition of the hair. The composition disclosed contains silicone lubricants, hydrolysed protein for strengthening the hair and lanolin moisturisers. The formulation was discussed in detail in chapter 2.

3.1.5 Research hypothesis of current chapter

This section of the study hypothesized that using a tool, typical to those described in the patents , or a similar item, alongside a formulation, typical to a commercial braid remover and such as that described in ZA~ 31 2010/04284[7] ,would decrease the strain or effort [force] of detangling braids by decreasing the frictional forces in the braid. The frictional forces can be decreased by increasing the lubricity of the hair within the braid by deposition of lubricants of the formulation onto the hair shaft. Many workers have studied the use of formulations containing cationic surfactants [8]. Cationic surfactants, as found in hair conditioners, have the ability to decrease the frictional properties of human hair. This is achieved by a deposition of the positively-charged surfactant onto the negatively-charged hair surface. The formulation under study is hypothesized to affect the human hair-to-human hair and the human hair-to-manufactured hair interactions. The tool used in this study was a comb.

3.2 EXPERIMENTAL DETAILS

3.2.1 Hair Samples

Dark brown hair samples were obtained from a 24 year old African female. The hair had a history of chemical treatment (relaxing). Manufactured hair samples were taken from a Kanelokan synthetic hair bundle supplied by YIWU Kanelokan Wigs Co. Ltd. The described hair samples were used for all the measurements in this chapter. The two formulations under investigation are shown in table 3.1 below. The control for the experiment was untreated braid samples. Two replicates were measured for both the control and formulation A. Four replicates were measured for test formulations B. The results of the treated braid samples were then compared to the results of the untreated samples in order to evaluate the differences in the detangling forces arising from the treatments.

Table 3.1 Hair treatment formulations (test formulations).

Ingredient	**% Formulation A**	**% Formulation B**
Cyclopentasiloxane	0	25
PEG-12 Dimethicone	0	5
18-Methyl Eicasenoic Acid	10	0
Isopropyl Palmitate	3	3
SETAKOL®	5	5
Hydrolastan	5	5
Hydroxy ethyl cellulose	0.2	0.2
Cetyl trimethyl ammonium chloride	2.50	2.50
Lanolin	0.15	0.15
Oleth -5	10	10
Olive oil	5	5
Water	q.s	Qs

3.2.2 Pre-treatment of human hair before braiding

The cleaning and preparation of the human hair tresses before they were braided was carried out as follows: A tress of weight 0.07 ± 0.001g was soaked under warm (40^{0}C) running tap water for 30 seconds. Each tress was then soaked and lathered with a 1ml solution of 12.5% Sodium lauryl sulphate (SLS) for 60 seconds in order to remove all dirt that may be present on the hair surface from previous treatments. The SLS solution was then thoroughly rinsed off the tress with warm tap water for 60 seconds. For drying, the hair tresses were hung under a Thermal Ionic Babyliss hood dryer set at heat setting no. 3 (~60 ^{0}C) for 15 minutes.

3.2.3 Braiding of samples

Braid samples were assembled manually using 0.07 ± 0.001g of chemically treated human hair and 0.5 ± 0.01g of synthetic hair as shown in figure 3.8.The synthetic hair was bent in half in order to create two sections of the braid entity. The length of all human hair used was 150 ± 10 mm and made up the third section. Although not entirely possible, effort was made to assemble the braid identically for each braid sample. For the type of braid that was studied, the human hair would be represented by the middle string in fig. 3.8.

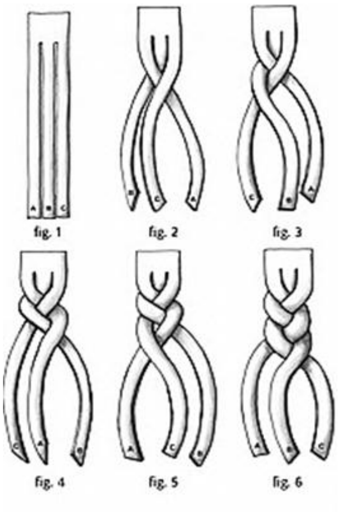


Figure 3.8 A Sketch of a three-way braid.

3.2.4 Treatment of braid samples with test formulations.

For the treatment of the braids with the test formulations, each braid sample was first soaked in warm running water for 30 seconds and then formulation A or B was applied along the length of the braid to saturate it for 60 seconds. The braid sample was then hung to dry under a hood dryer (temp ~60 ^{0} C) for 30 minutes without rinsing off the test formulation. The control samples were only braided and the combing forcing measured without any treatment.

3.2.5 Research Methodology

The research hypothesis was evaluated using a customized comb-out method from the well-known literature method by Garcia & Diaz [8]. Braided hair samples were detangled by combing them out. The combability of the braid samples, obtained by measuring combing forces, reflects the force required to detangle the braided samples. Garcia and Diaz define combability as the relative ease or difficulty with which human hair can be combed [8]. For the study, this definition was adopted for the detangling of braid samples; the detangling of braids can thus be defined as the ease or difficulty with which a braid can be detangled. The act of detangling is defined as the combing, getting knots out of and disentangling of hair. Garcia and Diaz's comb out method was adapted to best simulate the braid removing process by removing all the teeth, except one in the middle section of the combs used. Removing the teeth at the mid-section of the comb allowed for easier braid removal because the braid would get caught on the surrounding teeth. The set-up that Garcia and Diaz used is shown in figure 3.9. Later works by Kamath and Weigmann [9] use hard rubber combs instead of the stainless steel comb used by Garcia and Diaz. An earlier study conducted by Newman et al in 1973 also documents the use of hard rubber combs [10].

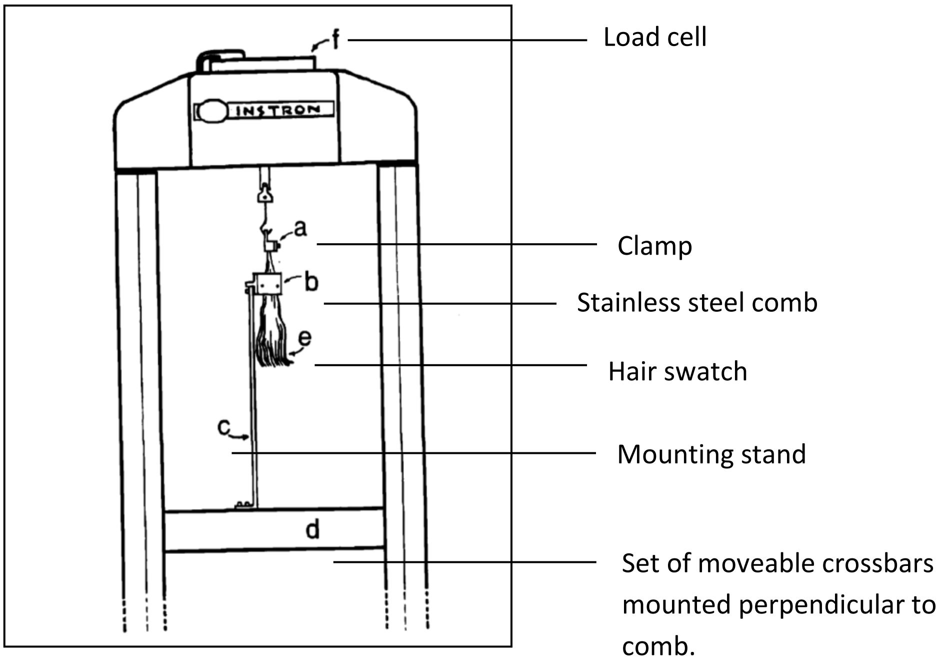


Figure 3.9 The set-up used by Garcia and Diaz to measure the combability of human hair [8].

Fig. 3.10 shows the experimental set-up used by Kamath and Weigmann, fig.3.11 shows the method that Newman et al used.

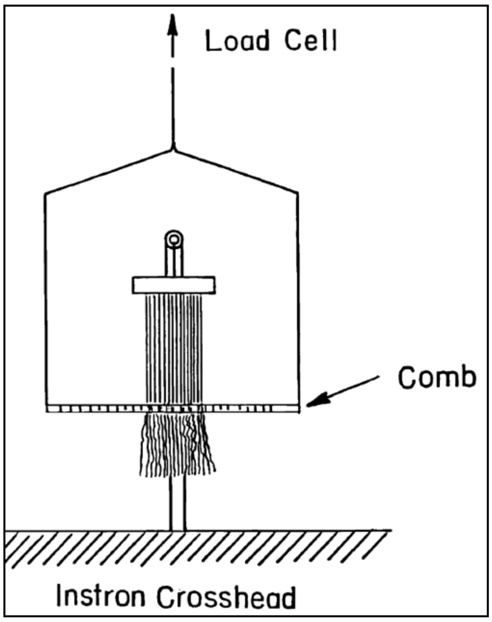


Figure 3.10 The experimental set-up used by Kamath and Weigmann to measure the combing forces of hair tresses [9].

Kamath and Weigmann preloaded the load cell and the combing forces were measured by observing a reduction in load as the hair tress was pulled through the comb at a rate of 8.3 mm/s [8] . Their method involved the measurement of the force required to pass a comb through a hair assembly. This measured force was then used to compare the conditioning effects imparted by different formulations.

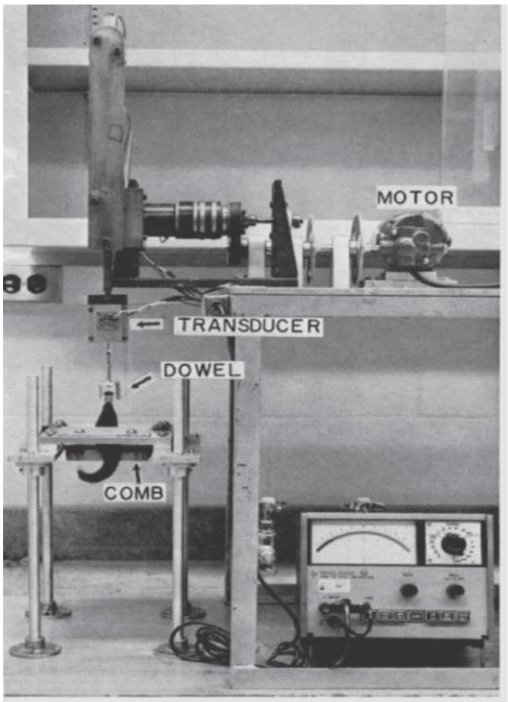
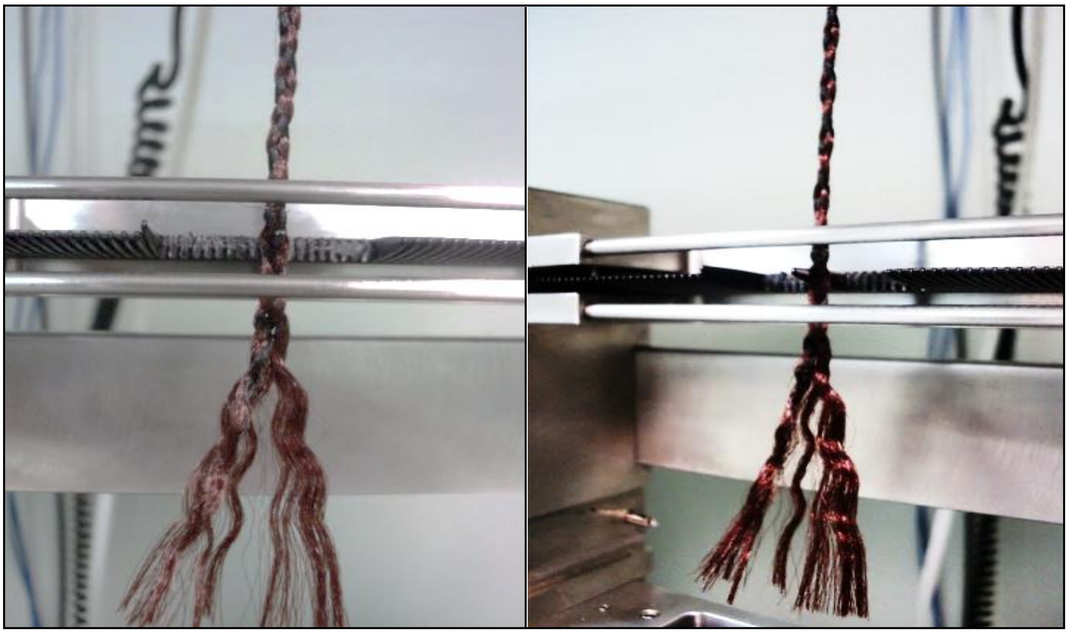


Figure 3.11 The apparatus used by Newman to measure combing forces [10].

The combability measurement methods discussed by Newman el at, Garcia and Diaz and other workers is based on measuring the ease or difficulty of combing human hair before and after it has been treated with different hair conditioning products or actives. This method is widely used in the research field and for the validation of hair conditioning claims in the hair care industry [9, 11]. For this study, braided hair samples were treated with two formulations containing similar ingredients as those used in hair conditioners and in other braid removers (see table 3.1). The 2 ingredients which were varied were a silicone mixture and 18-Methyl Eicasenoic acid (18-MEA).Formulation A contained 0% silicone mixture and 10% 18-MEA, whilst Formulation B contained a 30% silicone mixture and 0% 18-MEA.

Removing all the teeth in the middle section of the comb, except one, created a similar tool as those disclosed by inventors of braid detangling tools. A hard rubber comb was used. This set-up, as shown in figure 3.12 simulates the braid removing process where a sharp-toothed object like a safety pin or a fine-toothed comb is used to detangle the braid. As already explained earlier, the method used for measurements of the braid detangling forces was adapted and done in the same manner as the measurement of combing forces described by Garcia and Diaz [8] and by Kamath and Weigmann [9].



(a) (b)

Figure 3.12 A close-up image of the adapted comb out method used in this study. (a) is the front view and (b) is the side view.

Figure 3.12 and 3.13 is the experimental set-up that was used in this study. The braid detangling measurements in this study were made on an Instron housed at the Textile Research Institute, Princeton. For the experimental set-up, a braid sample was mounted on the load cell of an Instron 5500R tensile tester as shown in 3.13. A hard rubber comb with one remaining tooth in the middle section was inserted 5-10mm away from tip (end) of the braid in order to detangle that section of the braid. The detangling of the braid was started from the bottom of the braid and worked up, in the same manner that braid wearers remove their braids. The braid was detangled from the bottom, in ± 10 mm sections at a time. Each ±10mm section was labelled as a “plait”.

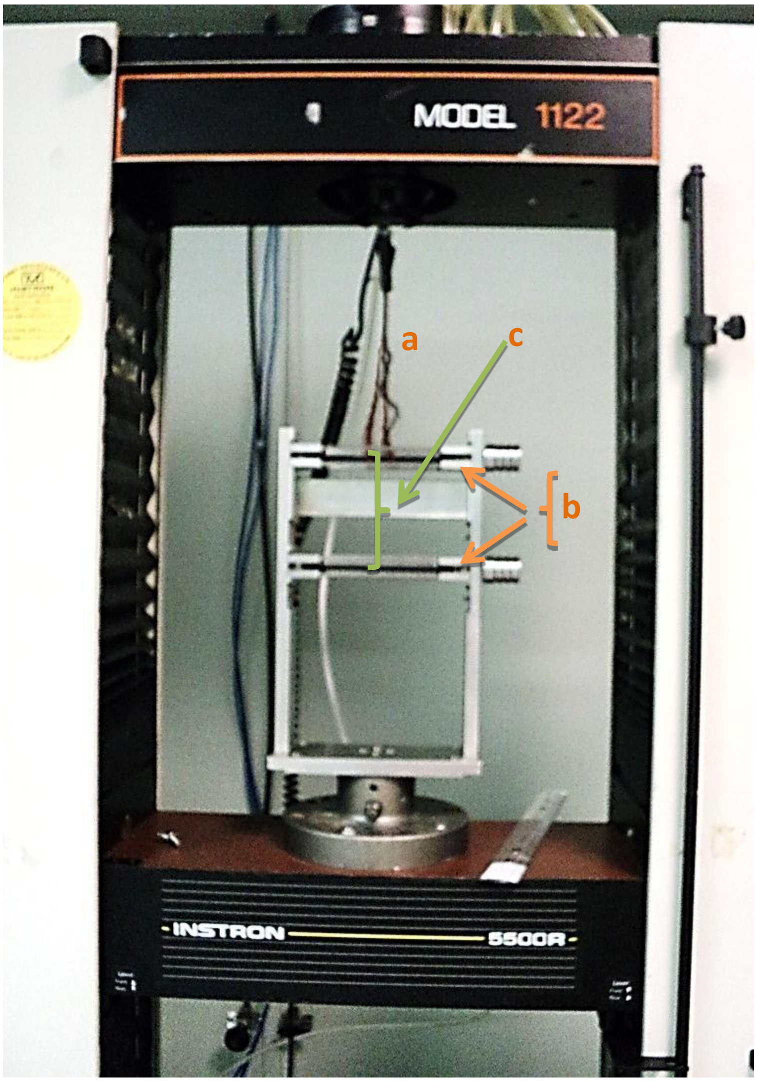
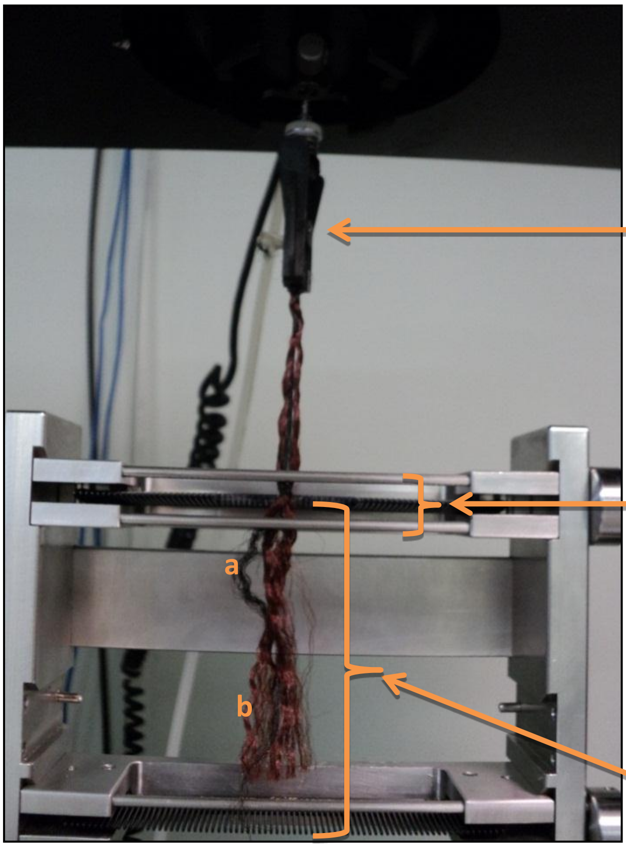


Figure 3.13 The Instron set-up used in the current study to measure detangling forces or force of detangling.

A smooth Aluminium retaining bar was used to prevent the braid (a) from slipping off the comb (b) during measurements. The top comb (c) was pulled through the braid at a rate of 2.5 mm/s. The bottom comb was not used in this study. The experiment was conduct in a controlled environment of T= 23 ± 1˚C and Relative humidity= 63 ± 1%. The force of detangling was measured in Newtons and this was plotted against the length of the braid sample. A close-up image of the experimental set-up is shown in figure 3.15. The comb was cleaned after each measurement.



Load cell measuring the force required to detangle the braid sample as the comb travels down the length of the braid. The force measured was plotted against the plait (5-10mm section).

Smooth retaining bars to prevent the braid sample from slipping out of the comb during detangling.

Two hard rubber combs. The braids samples were detangled using only the top comb.

Figure 3.14 A close up image of the experimental set-up at the textile research institute.

In figure 3.14, the human hair is the darker swatch of hair (a) and the manufactured hair is the brown swatch (b).

3.3 RESULTS AND DISCUSSION

This section presents the results obtained in the study as well as brief discussions for the observed trends.

3.3.1 Data Analysis

A series of t-tests and ANOVA's were used to test for significance of the results that were obtained in this study.

Definitions of statistical terms used:

1. **P-value:** When a null hypothesis (Ho) is proposed, such as Ho: $\mu_A = \mu_B$, the p-value is the probability to reject the null hypothesis unjustified.

Rejecting a null hypothesis unjustified is referred to as a Type I error. The null hypothesis is rejected when the p-value is below a selected significance level, α. The selected significance level chosen for all statistics in this report is 0.05. Rejecting a null hypothesis means that the results observed in a particular experiment are statistically significant. Accepting the null hypothesis therefore means that the results cannot be shown to be statistically significant for the experiment or observation

2. **Upper and lower confidence limits (UL and LL)**: Based on the assumption of normality, confidence limits for average values can be calculated. A 95% confidence interval for an average will contain the true mean with 95% confidence. The formula used to calculate confidence intervals is as follows:

$$\boldsymbol{UL} = \boldsymbol{X} + \boldsymbol{t_{\alpha/2}^{n-1}} * \frac{s}{\sqrt{n}} \text{ and } \boldsymbol{LL} = \boldsymbol{X} - \boldsymbol{t_{\alpha/2}^{n-1}} * \frac{s}{\sqrt{n}} \quad \text{(Eqn 3.1)}$$

Where X = average observation or result, also referred to as the sample mean

t= test static

n= number of observations

α = significance level

s = standard deviation

3.3.2 Detangling forces of braids treated with test formulations.

The force applied in detangling the braid sample was obtained for the three different samples: formulation A and B and a control. These formulations were discussed earlier in paragraph 3.2.1. The summary of the results of force (N) of detangling across the entire braid length are shown in table 3.2 below.

Table 3.2 Summary of the mean detangling force (in Newtons) results for the different treatments.

Sample	Control		Formulation A		Formulation B			
	1	2	1	2	1	2	3	4
Sample mean (N)	3.96	4.33	4.37	3.97	4.45	4.42	5.21	3.94
Std. Dev[1]	2.32	1.95	1.85	1.27	1.16	1.36	1.31	2.04
(95%CI) LL[2]	2.30	2.94	3.38	3.29	3.78	3.67	4.42	2.64
(95% CI)UL[3]	5.62	5.72	5.35	4.64	5.12	5.18	6.00	5.24

1 : Standard deviation, the variability of all the observations from the mean .

2 and 3: The 95 % confidence interval lower limit and upper Limit show that the true average braid detangling force lies between the upper limit and the lower limit.

The biological nature of the samples and the manual assembling of the braids were anticipated to introduce variations in the results. These variations are seen in the variability of the average detangling forces within samples that were treated in the same way, or with the same formulation. This variation is also reflected in high values for the standard deviations of all the samples tested. The different results are discussed in the following sections.

3.3.3 The first null hypothesis: 95% Significance test for untreated braid samples.

The null hypothesis to test the significance of variations in the control samples was that "there are no significant differences in the true averages of the two control samples", in short hand notation the null hypothesis was Ho: $\mu_{control1} = \mu_{control2}$. Accepting this null hypothesis would also demonstrate the reproducibility of the experimental method. A one way ANOVA was used to test the hypothesis. A summary of the statistics is shown below in table 3.3.

Table 3.3 ANOVA: Single Factor

For control braids (untreated)

SUMMARY

Groups	*Count*	*Sum*	*Sample mean*
Control 1	10	39.6	3.96
Control 2	10	43.29	4.329

Source of Variation	*F-stat*	*P-value*	*F crit*
Between Groups	0.14	0.71	4.41

The null hypothesis was accepted; there were no statistically significant differences in the true average detangling force of the two untreated braid samples. The supporting p-value for the decision was 0.71 (95% Confidence interval). The F-statistic for the results was also lower than the F-critical, supporting the decision to accept the null hypothesis. Figure 3.15 below is a plot of the average braid detangling forces measured for the two samples.

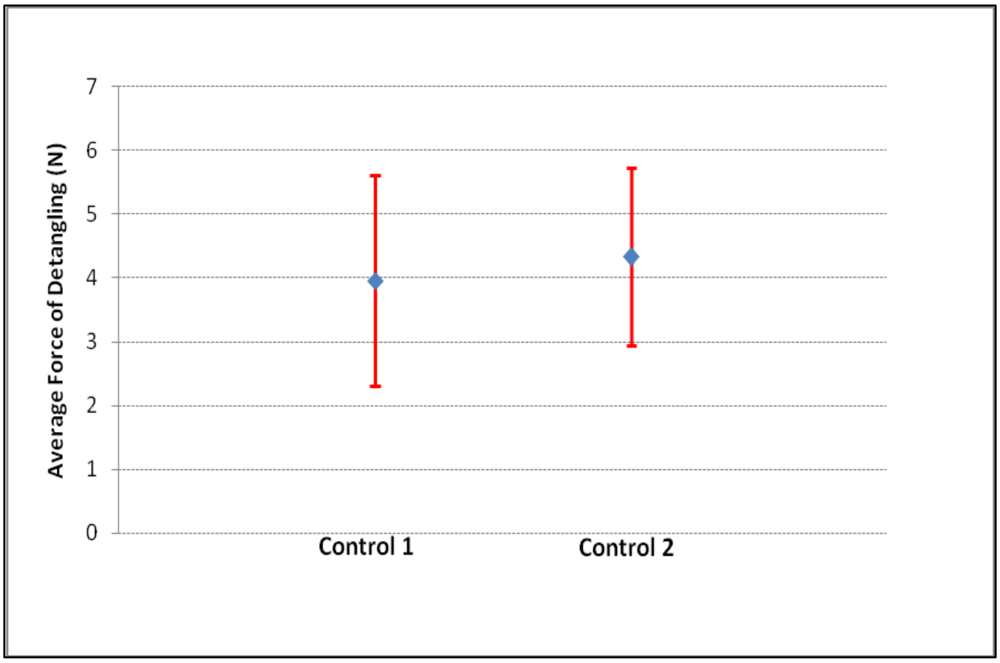


Figure 3.15 95% Confidence intervals for untreated braid samples.

Large confidence intervals were observed for both of the control samples, showing variations from the average detangling forces. Large variations in braid detangling forces were observed for the middle plaits, plait # 4 to plait # 9, see table 3.4 below.

Table 3.4 Measured Braid detangling forces for untreated braid samples.

	Braid Detangling Forces (N)	
***Plait #**	**Control 1**	**Control 2**
1	2.08	1.33
2	2.8	2.35
3	2.00	2.85
4	8.77	3.18
5	2.44	5.35
6	1.46	8.37
7	4.30	6.25
8	5.01	4.31
9	6.55	4.38
10	4.19	4.92
Sample mean	3.96	4.33
Std dev.	2.32	1.95

*Each plait represents a ±10mm braid section.

Figure 3.16 shows a graphical representation of the results in table 3.4. The variations in the mid-section of the braid can be seen more clearly. Plait 1 represents the end of the braided section of hair and plait 10 represents the braid section close the root of the hair. These variations are discussed in the next section.

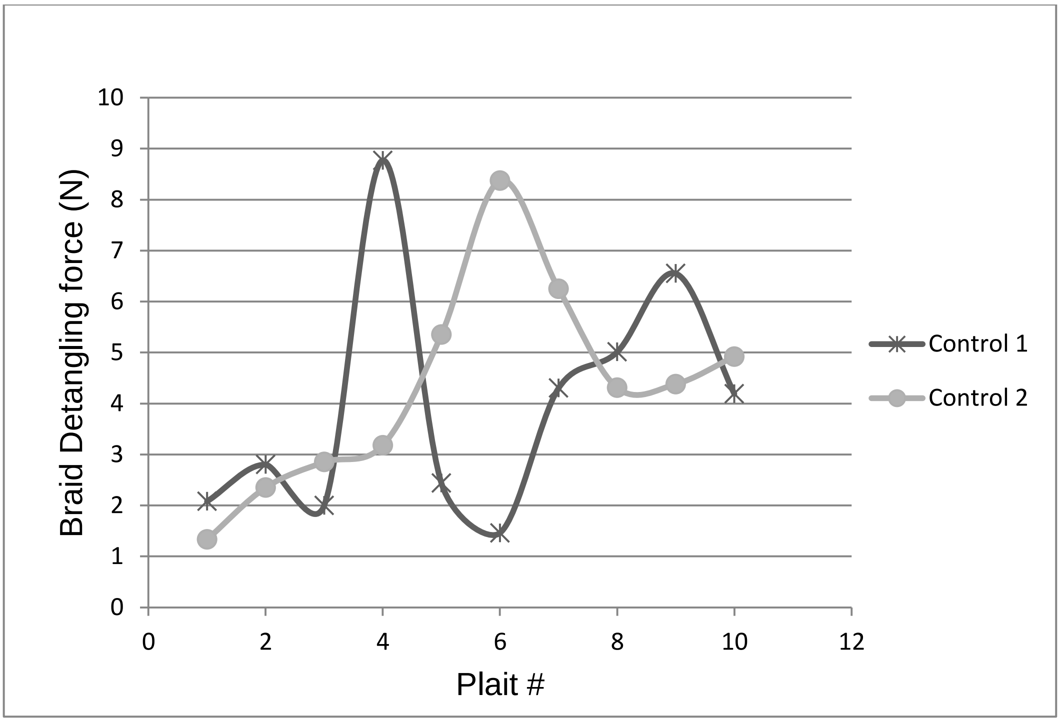


Figure 3.16 Observations of variations in the braid detangling forces of untreated braid samples.

3.3.4 The second null hypothesis: testing significant differences in braid detangling arising from different treatments.

In order to test if there are any significant differences in the braid detangling force for samples treated with formulations A and B, it had to first be proved that there no significant differences in the replicate treatments. This was done and summaries of the statistics are shown in figure 3.17 and table 3.5 for formulation A and in figure 3.18 and table 3.6 for formulation B. Two replicates were measured for formulation A and 4 replicates were measured for formulation B.

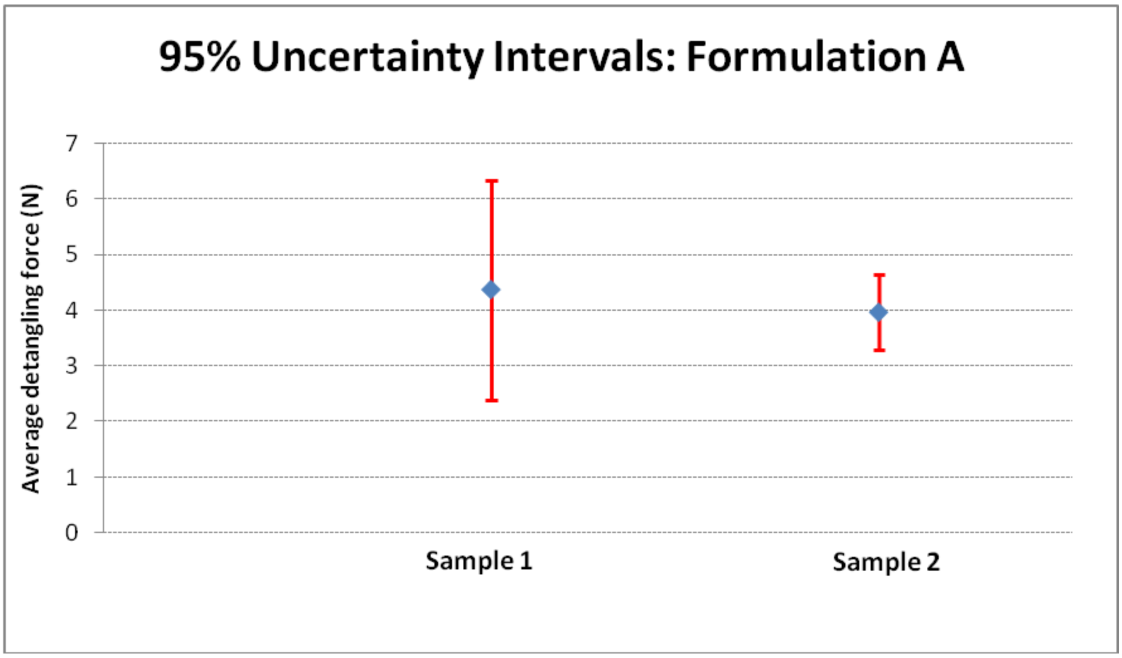


Figure 3.17 95% Uncertainty Intervals for braid samples treated with formulation A.

A t-test was conducted for the duplicate braid samples that were treated with formulation A. The second null hypothesis was accepted, viz: no significant differences were seen; the p-value obtained was 0.49 as shown in table 3.5.

Table 3.5 Formulation A replicates.

t-Test for significance :

	Sample 1	*Sample*
Sample Mean	4.37	3.97
Hypothesized Mean Difference	0	
test Statistic	0.71	
P-value	0.49	

In order to test if there were any significant differences in the true means of the four replicates for formulation B, an ANOVA was used. The null hypothesis tested was Ho: $\mu_1 = \mu_2 = \mu_3 = \mu_4$. The statistics can be found in table 3.6. No significant differences were observed, p-value=0.21. The true mean detangle forces of the four samples could not be shown to be significantly different. All the confidence intervals of the four replicates overlapped, supporting the conclusion that there are no significant differences between the four samples. Figure 3.18 shows a plot of the average detangling force of each of the four samples and their respective error bars.

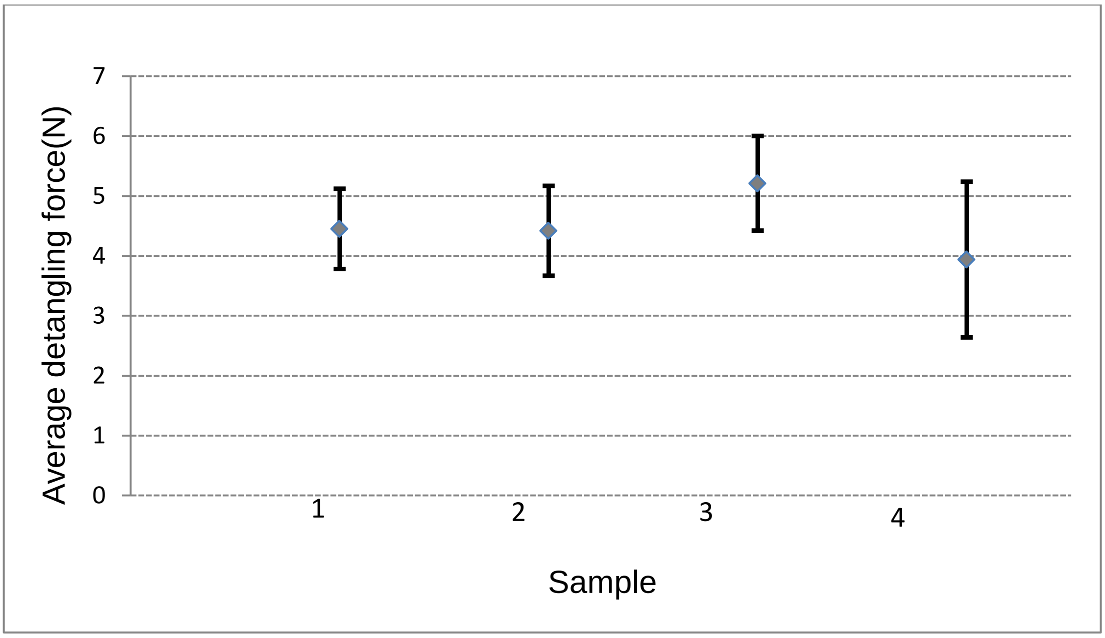


Figure 3.18 95% Confidence interval for braid samples treated with formulation B.

Table 3.6 Formulation B replicates.

Groups	*Mean*		
Sample1	4.45		
Sample2	4.42		
Sample3	5.21		
Sample4	3.94		
ANOVA			
Source of Variation	***F-stat***	***P-value***	***F crit***
Between Groups	1.56	0.21	2.79

Showing that there were no significant differences between the true mean detangling forces of the replicate samples of the same formulation then allowed for comparison of formulation A vs. formulation B. The hypothesis that was tested was whether or not there were any significant differences in the detangling forces of braids arising from the two test formulations. The data for each formulation was combined and statistical analysis was conducted on the two sets of data. The null hypothesis was as follows: "*<u>there are no significant differences in the true mean detangling force for braid samples treated with form. A when compared to the true average detangling force of braids treated with form. B</u>*." [Ho: $\mu_{formulation\ A} = \mu_{formulation\ B}$].This hypothesis was tested with a two sample t-test. The method did not pick up any significant differences in the detangling forces of braids; treated with formulation A when compared to those of formulation B. The p-value was above 0.05. A summary of the statistics obtained for the analysis is shown in table 3.7. Although it was expected that the presence of silicone lubricants in formulation B would decrease the detangling forces of braids treated with this formulation when compared to the braid detangling forces of samples treated with the formulation that contained no silicone; this was not the case. The data collected showed that no significant differences arise in the braid detangling forces measured for samples treated with formulation A or formulation B. The 95% uncertainty intervals for the two treatments overlap as can be seen in figure 3.19.

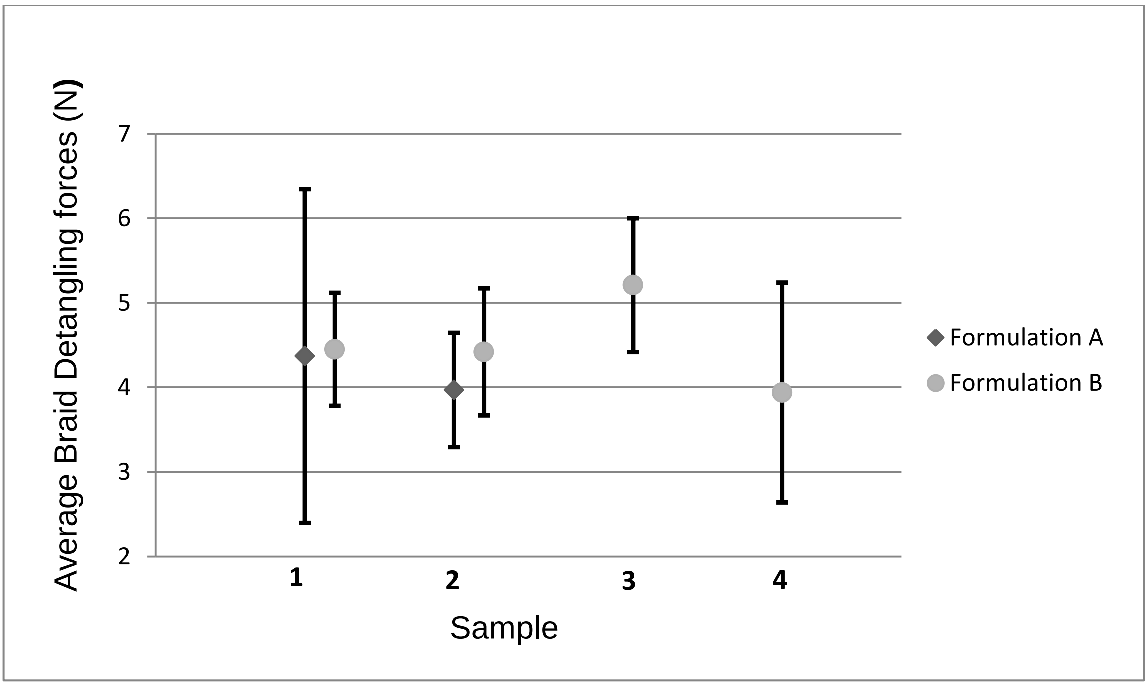


Figure 3.19 95% Confidence intervals for formulation A and formulation B.

Table 3.7 A summary of the statistics. Form. A vs. Form. B

	Formulation A	Formulation B
Sample Mean	4.17	4.68
Hypothesized Mean Difference	0	
P-value	0.133	

3.3.5 Third Null hypothesis: Treated braids versus untreated braids

The next step in the data analysis section of this study was to evaluate if there were any significant differences in the mean forces needed to detangle untreated braids when compared with braids that have been treated with form.A or form.B. Control samples were expected to exhibit higher detangling forces than the treated samples. The presence of silicone lubricants in formulation B was expected to decrease the braid sample's mean detangling forces. A one-way ANOVA was used to test for the significance with the following hypothesis: Ho: $\mu_{\text{untreated}} = \mu_{\text{formulation A}} = \mu_{\text{formulation B}}$. The finding however, was that there are no significant differences in the true mean force required to detangle the braids, whether untreated or treated with formulation A or B. A summary of the ANOVA output is shown in table 3.8. Figure 3.20 is a means graph of the untreated samples versus treated with 95% confidence intervals.

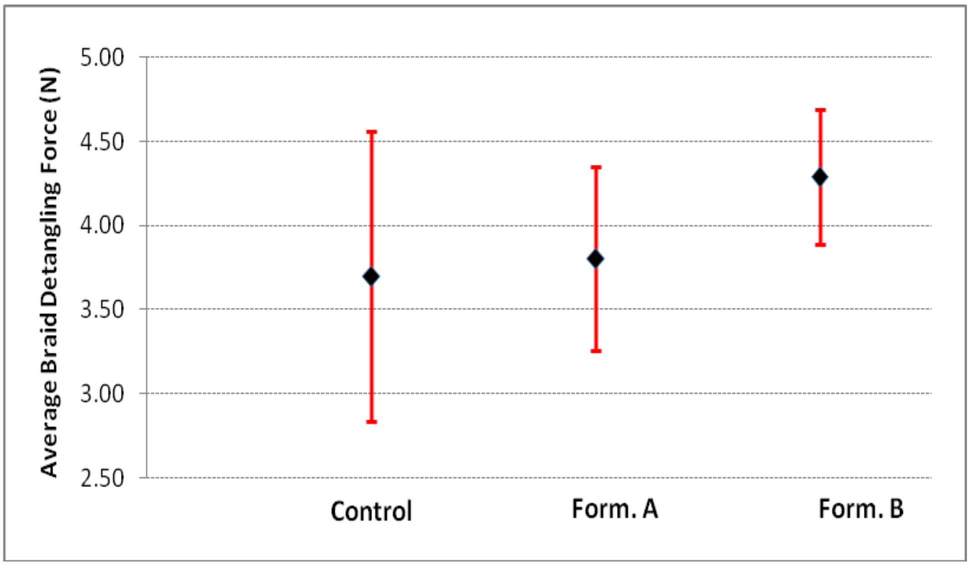


Figure 3.20 95% Confidence intervals showing the overlap in error bars for untreated samples versus treated samples.

The confidence intervals indicate that with 95% certainty, the true mean detangling forces of each sample lies between the upper limit and the lower limit. The confidence intervals for the three sample treatments overlap, showing that there are no significant differences in their true mean detangling forces. The p-value obtained was 0.55; see table 3.8 below, justifying the acceptance of the null hypothesis.

Table 3.8 Summary of statistical details of the 3 different treatments.

ANOVA SUMMARY

Groups	Mean Detangling force
Untreated	4.14
Treatment A	4.17
Treatment B	4.51

F-statistic	P-value	F critical
0.60	0.55	3.08

The conclusion for this set of data was that it could not be shown that there are any significant differences in the true mean detangling forces of the braids treated with a formulation containing lubricating silicone components or not. There are various proposed explanations to the above observations and conclusions:

1. The treatments were not adequately deposited onto the hair or synthetic hair to cause a change in the lubricity of the hair and a significant difference in the force required to detangle the braids. This can be as a result of insufficient treatment applied, or insufficient deposition time or merely the nature of the braid. The intertwined nature of the braid made it difficult for the treatment to deposit uniformly onto the hair shaft. Conditioner treatments that are administered onto the hair shaft deposit in areas when the hair cuticle is fragmented. When applied onto the hair shaft, conditioners thinly coat the hair via Van der Waal's forces of attractions [12]. The test formulations under investigation in this study had the same attributes that hair conditioners have. Hair conditioners contain cationic surfactants, fatty alcohols and water. These three functional actives were present in both of the test formulations. Hair conditioners work by forming a thin coating on the surface. This layer is said to be able to change the tribological properties

of the hair's cuticle surface [12]. Figure 3.21 and figure 3.22 show how La Torre and Bhushan [14] illustrated the localization of positively charged conditioners onto the negatively charged hair shaft (see figure 3.22).

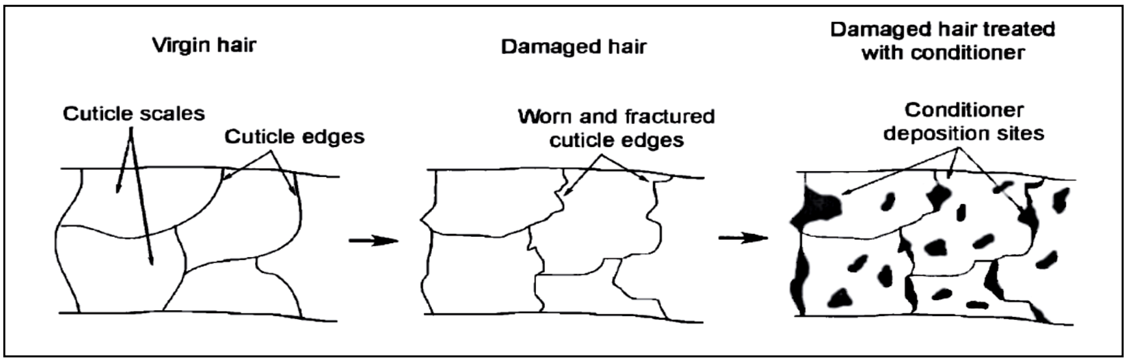


Figure 3.21 The deposition of conditioners on the hair cuticle surface.

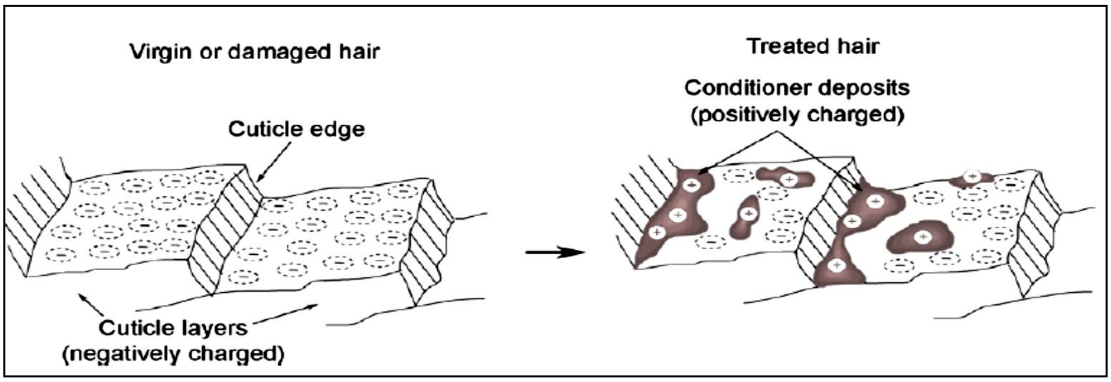


Figure 3.22 An illustration of how positively charged conditioner deposits onto the negatively charged hair cuticle surface.

SEM imaging was done on hair from treated braids in order to see if there was any visible deposition of the test formulation on the hair shaft surface. The images are shown below in figure 3.23 and figure 3.24.

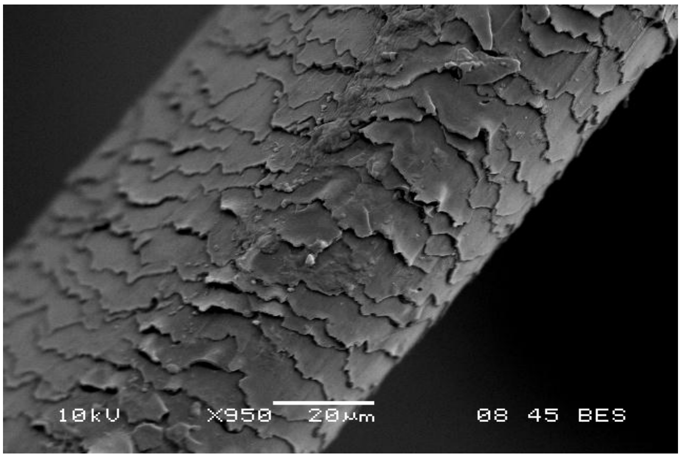


Figure 3.23 SEM image of hair strand taken from a bundle of untreated hair.

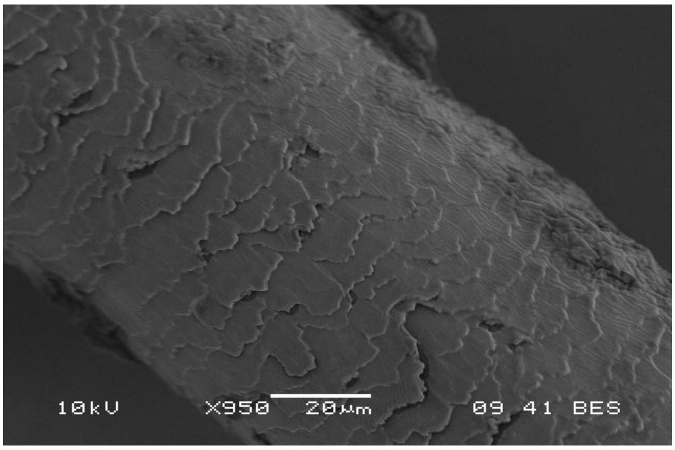


Figure 3.24 SEM image of a hair strand taken from a braided hair swatch that has been exposed to a test formulation.

The SEM imaging of treated hair samples showed a film of lubrication on the hair surface, as can be seen in figure 3.24.

According to La Torre and Bhushan, a conditioning formulation tends to accumulate on the cuticle edges more than on the rest of the hair shaft. This principle was not seen in the localization mechanism for the test formulations. Having the hair in braids is thought to have made difficult for the treatment to reach these deposition sites and cause any significant effects in the lubricity of the hair shaft.

2. The other proposed explanation as to why no significant differences were seen in the results is that the braid detangling is not influenced solely by hair friction but also by the make-up of the braid. This means that, the presence of a lubricating formulation may decrease hair friction to some degree, but this decrease is overpowered by the simple nature of the braid. How the braid is done; the

tightness of the braid , the synthetic hair used and the size of the hair swatch in the braid, were thought to be the primary influences affecting the ease of braid detangling. Although the study was unsuccessful in decreasing the combability force of braided samples, the method developed herein can be used for further work on braids.

3.3.6 Differences in the detangling forces of different sections along the braid length.

Although no significant differences were seen in the detangling efforts of treated braid samples, there were differences observed in the detangling forces in the different regions along the length of each braid. In the collection of all the data, the braid sample was demarcated into three sections because of the physical make-up of the braid. The braid detangling forces were measured from the tip of the braid to the top of the braid. Section 1 was the section towards the tip of the braid. Section 2 was the middle of the braid and Section 3 was the section closest to the root of the hair.

Section 3 is demarcated as the section that would be closest to the scalp if braids were on a wearer’s head. This section contains the thickest hair because hair is thickest close to the scalp and tapers off and is thinnest at the ends [13]. For braid samples containing 150 ± 10mm of human hair, this section was measured to account for 80 - 100 mm of the braid.

Section 2 is the middle section of the braid length. In this section, the human hair is less thick. The thinning of the hair swatch results in irregular braid strands. In order to remedy this irregularity, the synthetic hair is distributed equally and integrated into the thin human hair. This integrated swatch is then separated again into 3 strands and the braiding is continued. This ±30 mm section of braid was observed to have severe knotting because of the integration of human hair into manufactured hair.

Section 1: This last braid section, the tip of the braid, contains the thinnest hair. The bulk of the braid in this section is made up of the manufactured hair. This section accounted for approximately ±20mm of the braid length.

When the detangling forces of the braids were measured, it was observed that section 2 yielded the highest braid detangling forces. This was attributed to the severe knotting of human hair and synthetic hair in this section. The observations made in these sections are discussed in the next section.

3.3.6.1 Data Analysis of braid sections

There were significant differences observed in the detangling force of section 1 when compared to both sections 2 and 3 .The 95% error bars of section 1 vs. section 2 and section 1 vs. section 3 did not overlap for untreated braid samples as shown in fig. 3.25. Section 1, the section at the tip of the braid, with the least amount of human hair, showed the lowest average measured detangling force.

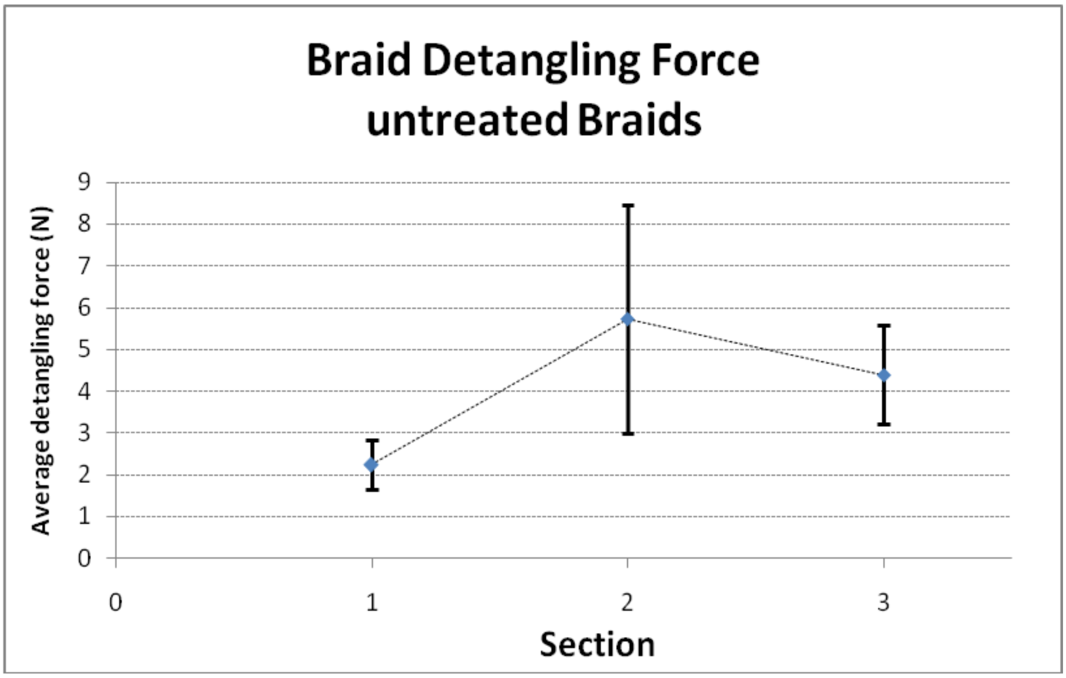


Figure 3.25 Braid Detangling Forces: Different sections along the braid length.

For the untreated braid samples, the mid-section showed the highest level of variation, as seen by the wide confidence intervals. The graph shows a plot of the average detangling force measured in the three braid sections. It was observed that the highest detangling forces were generally in the mid-section of the braid sample. The graph above in figure 3.25 shows that section 2 had the largest detangling forces. A one-way ANOVA was conducted on the data sets for the control (untreated) samples in order to ascertain the statistical validity of these observations. The results are shown in table 3.9 below.

Table 3.9 A summary of the significant differences in the braid detangling forces of the demarcated braid sections: Control samples

Groups	Average	Variance
Section 1	2.24	0.32
Section 2	5.73	6.7
Section 3	4.39	2.00

F-stat	P-value	F crit
6.41	0.0084	3.59

The null hypothesis proposed that there were no significant differences in the true mean braid detangling forces along the braid: $\mu_{\text{section 1}} = \mu_{\text{section 2}} = \mu_{\text{section 3.}}$ This null hypothesis was rejected (p-value = 0.008). In order to evaluate which of the three sections was significantly different from the others, a series of t-tests were done. The results are shown in table 3.10.

Table 3.10 Significance differences of mean detangling forces in untreated braid sections by t-Test.

Section 1 versus Section 2	Section 1 versus Section 3	Section 2 versus Section 3
Significant	Significant	non-significant

Section 1, with the least amount of hair, showed braid detangling forces that were significantly different from those found in sections 2 and 3. No significant differences were observed between sections 2 and 3. Section 2 had the largest sample mean detangling forces because of the intertwining of the synthetic hair and human hair in this section. Similar trends were observed for the treated braid samples; figure 3.26 and figure 3.27 show the average detangling forces of the three braid areas for the two test formulations.

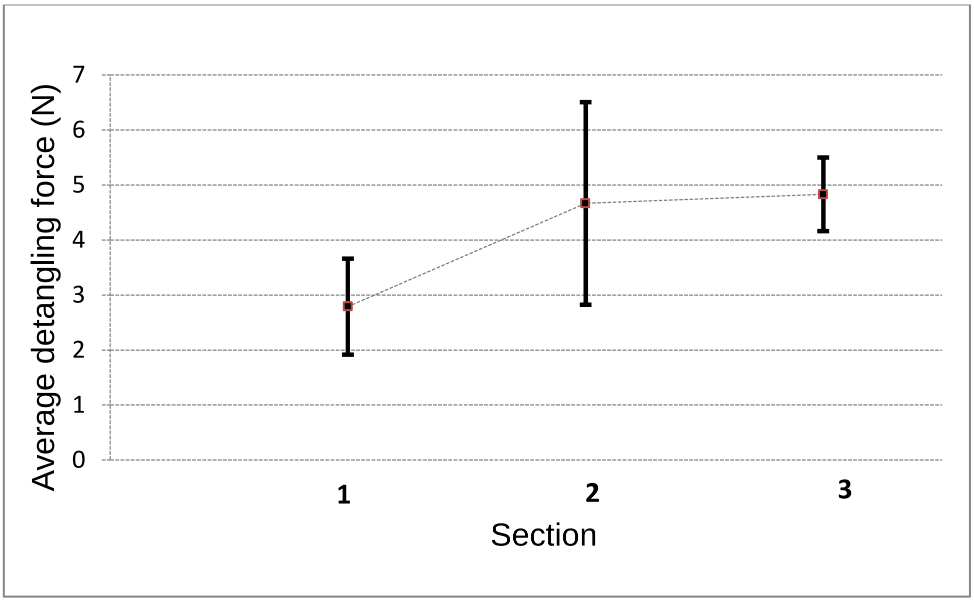


Figure 3. 26 Mean braid detangling forces along braid samples treated with formulation A, Section 1 was showed to be significantly different from section 3.

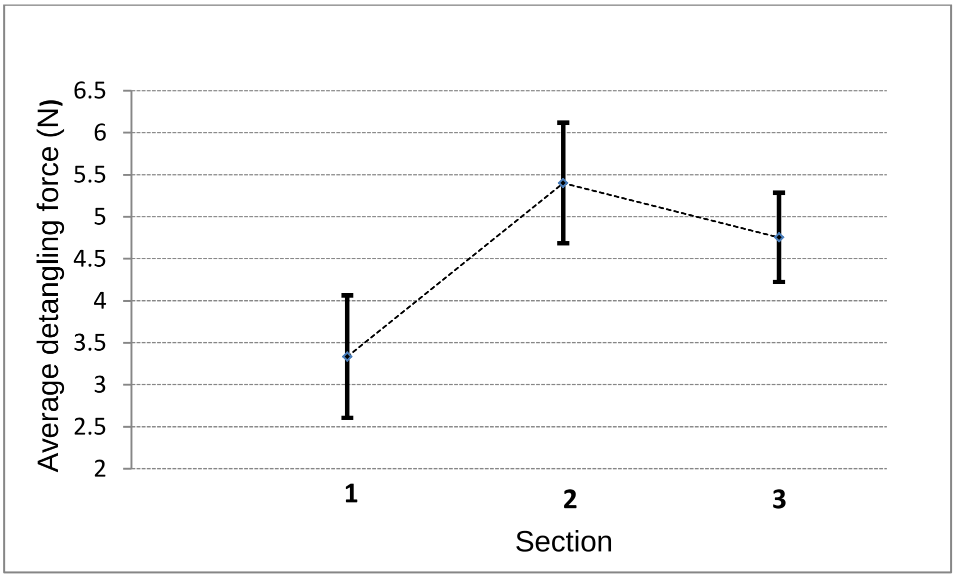


Figure 3.27 Average braid detangling forces along braid samples treated with formulation B.

The observations that were made for all the braid samples were combined. This could be done because it was proven earlier in sections 3.3.3 -3.3.5 that there were no significant differences in the true mean braid detangling forces for the three sample settings. The pooled observations were sorted according to the three sections of each braid. Fig. 3.28 shows the 95% uncertainty intervals for the different braid sections. From figure 3.28, it can be seen that the uncertainty bars for section 1 did not overlap with those of section 2 or section 3. A one way ANOVA was applied to the observations to test for significance in the three sections. The summary of the ANOVA is tabulated in table 3.11 below.

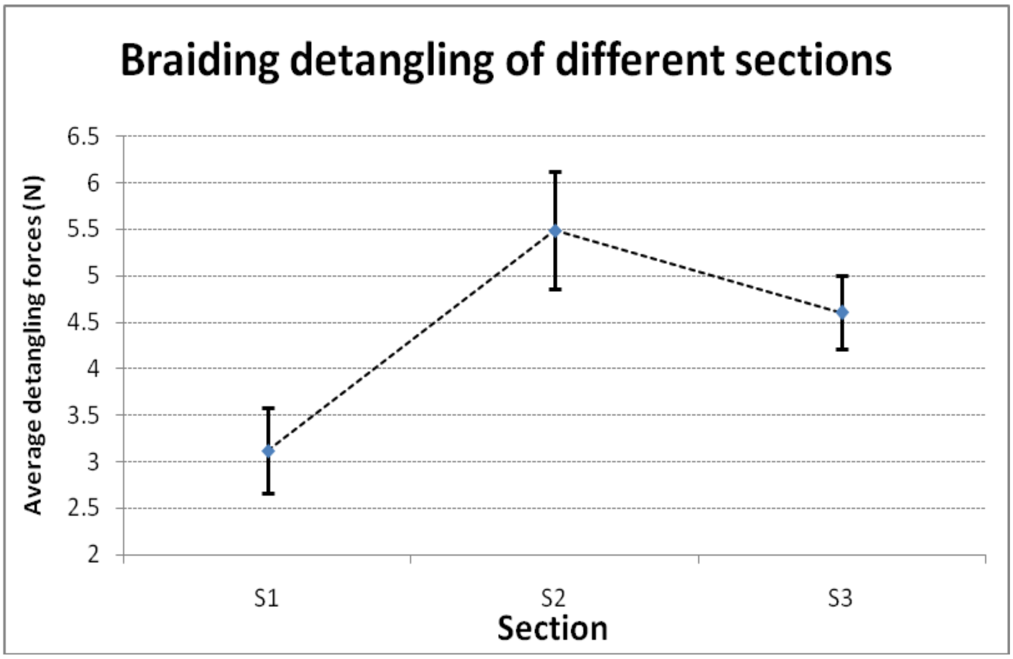


Figure 3.28 95% Confidence intervals of the three braid sections.

The uncertainty bars for S1 and S2 did not overlap, showing that there was evidence that the two sections differ significantly.

Table 3.11 ANOVA Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
S1	36	112.19	3.12	1.86
S2	33	181.16	5.49	3.12
S3	60	276.3	4.61	2.37

ANOVA

Source of Variation	**F-statistic**	**P-value**	**F critical**
Between Groups	20.87	1.49E-08	3.07

The null hypothesis for the pooled data set was as follows: Ho: $\mu_{s1} = \mu_{s2} = \mu_{s3.}$ This null hypothesis was rejected, p-value <0.00001. This supports the observation made from figure 3.28. This figure showed that the means of the three sections did not overlap. The error bars for section 2 overlap with those with section 3. A t-test was conducted on the data sets for section 2 and 3 in order to test for significance in these sections. The results for the t-test are tabulated below in table 3.12.

Table 3.12 Summary of significance test for section 2 and 3.

Two sample t-test

	S2	*S3*
Mean	5.49	4.61
Variance	3.12	2.37
Hypothesized Mean	0	
test Statistic	2.52	
test Critical one-tail	1.66	
P-value	0.0136	
t Critical two-tail	1.98	

The p-value obtained for the t-test was 0.01, thus the null hypothesis is rejected; the true mean detangling force of the two sections differ significantly. All the data analysis conducted in section 3.3.6 proved that although there are no significant differences arising from the treatments applied onto the braids, the method was able to show that there were significant differences in the force required to detangle various sections along the braid length.

3.4 SUMMARY AND CONCLUSIVE REMARKS

In this chapter, the combing forces of braids have been measured from the adaption of a normal combing instrument, using a widely used combing method. The results showed that the method did not pick up any significant differences in the braid samples that had been treated with test formulations and those which were untreated. There were significant differences in the braid detangling forces along the braid length arising from the braid make up.

Although the study was unsuccessful in picking up differences between treated braids and untreated braids, the method was able to measure significant differences in the detangling forces of sections along the braid. The method developed in this study can be improved and used to investigate the detangling of braided hair, which has not been reported in any scientific literature before.

3.5 SUGGESTED FUTURE WORK

- To minimise experimental variations, a braiding tool can be used to assemble the braid samples. This would eliminate variations arising from manual braiding.
- More braid samples should be tested for all treatment settings under evaluation.
- The absence or presence of the three braid sections on braids made with a braiding tool can be investigated.
- The presence of the three braid sections on braids made only with human hair can also be investigated.
- The process of formulation deposition on the braid can be improved.

3.6 REFERENCES

1. Taylor et al, inventor; Joseph Taylor, Brenda M Taylor. Debraiding tool. United States Patent 5071920. 1997 Dec 30.
2. Carty et al, inventor; Jennifer Carty, Kenneth Carty. Braid Detangling Device. United States Patent 5911225. 1997 Jun 15.
3. Robinson, inventor; Angela Mae Robinson. Tool for the removal of braids in hair. United States Patent 6095154. 2000 Aug 1.
4. Love-Johnson, inventor; Deborah Love-Johnson, Shawnee. Braid removal tool, United States Design Patent D6361225. 2011 April12.
5. Phillips, inventor; Alton Phillips. Braid removal tool. United States Patent 6021783.2000 Feb.
6. The Unbraider www.theunbraider.com(Feb 2010)
7. 2010/0428~ZA. Hair treatment composition.

8. Bharat Bhushan. Nanoscale characterization of human hair and hair conditioners. Progress in Material Science 2008; 53: 585 - 710

9. Mario L. Garcia &Jose Diaz. Combability Measurements on Human Hair. J.Soc. Cosmet. Chem.,1976;27:379-398

10. Y. K Kamath and Han-Dietrich Weigmann. Measurement of Combing Forces. J. Soc. Cosmet. Chem., 1986;37:111-124

11. Walter Newman, George L. Cohen and Cletus Hayes. A Quantitative Characterization of Combing Force. J.Soc. Cosmet. Chem., 1973;24:773 -783

12. Indira P. Seshadri, Bharat Bhushan. Effect of ethnicity and treatments on the in situ tensile response and morphological changes of human hair characterized by atomic force microscopy. Acta Materilia, 2008; 56: 3585 -3597.

13. Textile Research Institute Testing

14. http://www.triprinceton.org/testing/hair-testing-services/conditioning?pid=84 (last accessed Nov 2011)

15. Carmen La Torre, Bharat Bhushan, Jian-Zhang Yang and Peter M. Torgerson. Nanotribological effects of silicone type, silicone deposition level and surfactant type on human hair using atomic force microscopy. J. Soc. Cosmet. Chem. 2006;57:37-56

16. Clarence R.Robbins. The physical and mechanical behaviour of human hair. 3rd Edition. 1994 ,Springer-Verlag

CHAPTER 4

HAIR FRICTION AS AN INDICATOR OF HAIR LUBRICITY AND ITS *THEORETICAL* ROLE ON THE EASE OF BRAID DETANGLING

4.1 INTRODUCTION

4.1.1 Friction

Friction is defined as the force that resists motion when one body slides over another. The frictional force required to cause one surface to slide over another is proportional to the normal load that causes the two surfaces to press together [1]. Frictional force is depicted by the following equation:

$$\text{Frictional Force} = \mu N \qquad \text{(Eqn. 4.1)}$$

where N is the normal load pressing the two surfaces together and μ is the proportionality constant and is called the coefficient of friction. Laws of friction state that the frictional force of two surfaces moving over each other is independent of the area of contact between the two surfaces.

4.1.1.1 Coefficients of friction

In the sliding of two surfaces over each other, there are normally two kinds of forces involved; firstly, the force represented by μ_s, which is the minimum force required to start the sliding of the two surfaces. This force gives what is called the static coefficient of friction [2]. The second coefficient is called the dynamic or kinetic coefficient (μ_k) and is defined as the force necessary to maintain the sliding of the two surfaces after it has started [2]. For this study, only the dynamic coefficient of friction was studied. The friction between two surfaces sliding over each other is affected by lubricant materials that may be present between the rubbing surfaces [1, 2, 3]. The present work deals only with the friction of dry surfaces.

4.1.2 Frictional properties of human hair

The frictional behaviour of hair is mainly related to the cuticle.[1] The cuticle, which is the outermost region of the hair fibre, is important because it is this region that comes into contact with combing devices, skin and other fibres. The cuticle is a multi-layered region, made up of 5-10 flat layers of overlapping scales. The shape and orientation of these layers is what determines the differential friction effect in hair. Attached to the surface of the cuticle scale is 18–Methyl Eicasenoic Acid (18-MEA), a saturated fatty acid [4, 5]. This fatty acid contributes to the lubricity of the hair [4]. The surface of the hair fibre is negatively charged. 18-MEA is covalently bonded to the hair's outer surface via thioester linkages [5]. Figure 4.1 below shows how the 18-MEA binds to the epicuticle at the outer surface of a hair fibre.

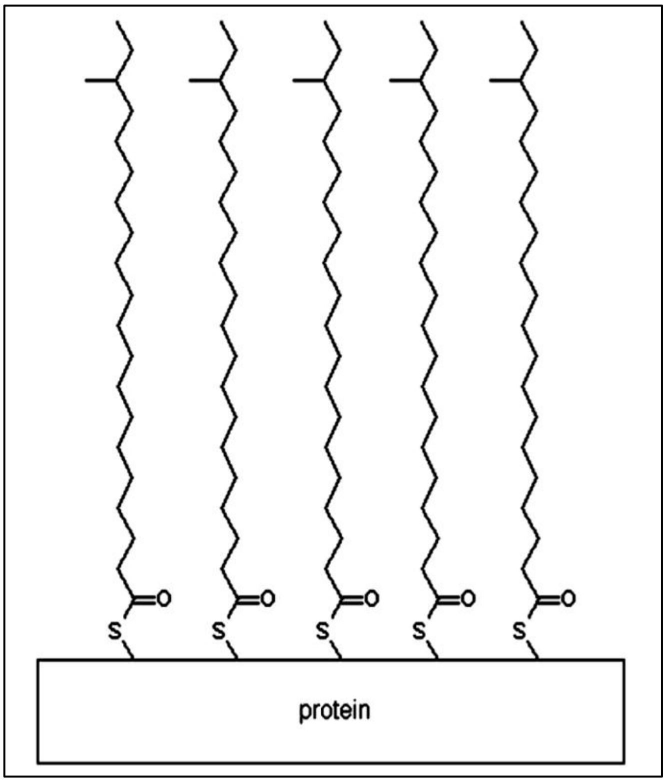


Figure 4.1 A schematic illustration of how 18-MEA binds onto the outer surface of a hair fibre.

4.1.3 Directional dependence of hair friction

Human hair has a directional effect and dependence. The hair fibres exhibit different frictional values depending on whether the fibre is rubbed from root to tip (R-T / along the hair shaft) or in the opposite direction of tip to root (T-R / against the hair shaft). It is easier to move a surface along the hair shaft than against it. This directional dependence is of importance in experiments involving the movement of multiple hair strands. Figure 4.2 below shows different scenarios of hair fibres moving against each other and the hair-to-hair interactions that would be present. Scenario A is the most common assembly of hair fibres on the head. The hair samples used in this study where aligned as shown in this scenario. This scenario represents the alignment of hair on one’s head where the root ends are anchored in the scalp. In scenario A, the scale edges of one of the fibres is rasping, no matter what direction the fibres slide over each other.

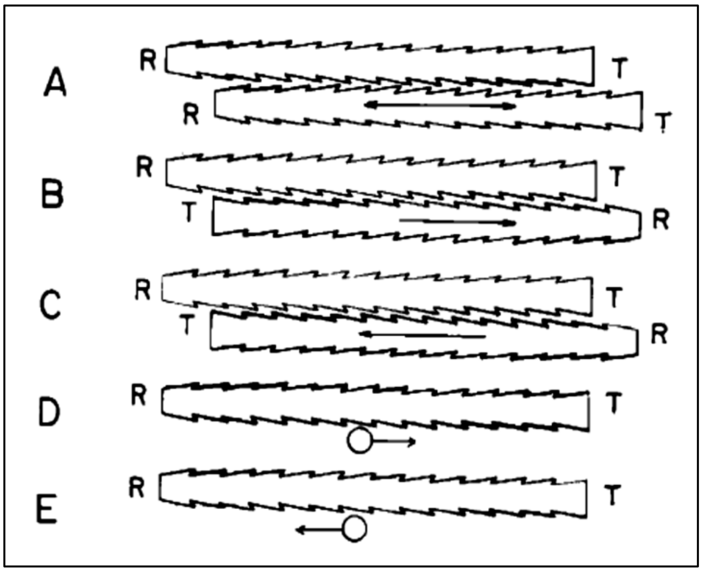


Figure 4.2 Directional effects in hair friction. The top hair strand is stationary, whilst the bottom strand is moving [2].

Other factors that affect hair friction are the physical properties of the two surfaces, the moisture content of the hair fibres, the working temperature for the experiment and the relative viscosity of the lubricant in the case of lubricated samples.

4.1.4 Hair damage and its effect on hair friction

Damage to the hair shaft can be inflicted by a variety of grooming techniques: permanent hair waving, chemical relaxation, colouring, bleaching and mechanical combing, and blow drying [1]. Repeated damage on the hair shaft from the above-mentioned techniques causes the negative charge on the hair to increase. An increase in the negative charges on the hair shaft causes increases in hair friction, adhesion and in the so-called static electricity ‘‘fly away’’. This increases the frictional properties between the hair fibres, making it harder to comb. Hair entanglement occurs more readily in damaged hair than it does in undamaged hair. Ali Syed, in his paper on hair damage; its causes, prevention and cures, investigates evidences of hair damage [6]. A summary of these evidences and prescribed remedies is attached herein as appendix 4.1. Of interest to this current study are the prescribed treatments for all the hair damage that Syed studied. Syed prescribes conditioning of the hair to be the remedy for moisture loss, brittleness, fly-away fibres and excessive tangling.

4.1.5 Treatment of damaged hair

The use of lubricating formulations, such as conditioners or conditioning shampoos, to coat the hair in order to prevent future damage to the hair is also reported by La Torre and Bhushan [7]. Conditioners coat the hair surface by Van der Waal’s forces of attractions. This coat is said to give rise to smoother and softer hair feel [4, 6, 7]. The softness and condition of human hair is judged by how the hair feels to the touch. The friction that is encountered when the hair is touched is dependent on the frictional forces of individual fibres and how they interact with those around them. These frictional forces then influence the overall friction of the head of hair and the hair’s manageability [2].

4.1.6 Background of study

In the development of a cosmetic formulation that would facilitate the process of detangling braids from chemically treated hair, hair friction was amongst the factors that were thought to influence ease of braid detangling. The overall aim (discussed in detail in chapter 1 and 2) of this dissertation was to investigate some of the factors, other than braid tightness, that affect the ease with which braids are detangled from the human hair.

These factors were identified to be:

1. Hair lubricity/condition
2. Hair strength
3. Oil-build (matting)

The above factors can be altered by treating hair with chemical formulations that contain ingredients that are said to have the ability to significantly improve these factors. In the current chapter, an attempt is made to decrease the frictional forces within the braid by adding a lubricating formulation between the human hair and the synthetic hair.

4.1.7 Hair friction and braided hair

Hair friction and adhesion are important indicators of hair feel and how the hair will interact with combs, skin and materials that it comes into contact with. Hair friction is an indicator of hair condition. In hair braiding, hair friction is reasoned to have an influence on the ease of detangling the synthetic hair that makes up the braid from the human hair. Lubricants that are present between two surfaces that are sliding over each other affect the frictional forces between the two surfaces [2].

After no significant differences were found in the ease of detangling of braids that had been treated with formulations containing lubricants (see chapter 3) because of, possibly, the insensitivity of the method or the complex make-up of the braid, a narrower study was proposed. This study investigated the frictional forces arising from interactions between the human hair-to-synthetic hair. These are the interactions that would be present in the braid, without the additional influences arising from the manner in which the braid is assembled. The most significant of these additional factors is the pressure applied to the hair when braiding. A swatch of human hair was caused to slide over a section of synthetic hair and the frictional forces were measured. Because of the directional dependence of friction, the hair swatches were moved from root to tip, the direction of the lowest friction. The friction arising from these interactions is thought to be a representation of the frictional forces that would influence whether or not synthetic hair can simply be “eased off” in the braid detangling process. Table 4.1 is a summary of some of the other factors that were thought to have an influence on the ease of braid removal. These were discussed earlier in chapter 2.

Table 4.1 Factors influencing ease of braid detangling.

Factor influencing braid detangling	Effect of factor
1. Braid size	Thicker braid = easier removal, less time
2. Force exerted in braiding process	Tighter braid = harder detangling
3. Texture/ condition of hair	Silkier hair = easier braid removal
4. Length of hair within braid	Longer = harder or more time consuming
5. Type of manufactured hair used	Silkier hair = easier detangling
6. Hair moisture content	Dry = harder, hair more brittle
7. Dandruff & oil build-up (matting)	Matted braid = harder (newer / fresher braids are easier to remove than older braids)
8. Braided region	Hair line is hardest to detangle because care must be taken because hair is weakest in this region [8].

It was observed that it generally seems somewhat easier to detangle braids from silkier hair than from kinky African hair. Moreover, it was also observed that it is easier to detangle braids from chemically altered (relaxed) African hair than from virgin African hair. From this observation, premature though it may be, it was concluded that the kink in African hair has some effect on the ease of detangling braids. Caucasian hair has a straight, smooth structure, whilst African hair is a cylindrical coil with kinks [1].

4.2 RESEARCH HYPOTHESIS

The research hypothesis for this part of the study was as follows:

"Introducing a lubricating formulation between the human hair and the synthetic hair that make up the braid will decrease the friction between the two surfaces."

The presence of a lubricating formulation is hypothesized to soften and "slicken' the braid and hair combination, allowing them to be detangled with ease.

Testing the research hypothesis:

- Increase the lubricity of human hair by applying a cosmetic formulation.
- Measure the sliding friction as human hair is caused to slide over a section of synthetic hair.

4.2.1 Research Objectives:

1. To develop a method that can reliably and reproducibly measure hair friction.
2. To increase the lubricity of hair in order to allow for easier braids detangling.
3. To optimize the lubricity of the formulation.

4.3 EXPERIMENTAL DETAILS

4.3.1 Development of experimental method

The method used in this study was adapted from a method discussed in G.V. Scott & C. R. Robbin's 1980 article entitled "Effects of surfactants on hair fiber friction" [1] and from an approach employed by Schwartz and Knowles in an earlier study [2,9]. The article by Scott & Robbins documents the effects that anionic, cationic and amphoteric surfactants have on the friction of a hair fibre. To quantify these frictional effects on human hair, the above researchers used a Capstan method [2, 9] and Roeder's method [10]. The Capstan method measures the forces required to slide a weighted hair fibre over a curved surface of reference material [9] .The curved surface is often referred to as a mandrel [1, 2 and 9]. The method by H.L Roeder measures the friction of a single fibre on a bundle of similar fibres [10]. This method can be customized for friction measurement of hair tresses. Figure 4.3 shows a schematic diagram of the customized Roeder method. Roeder used the method to study the frictional properties of textile fibres but the method can be used for human hair as well.

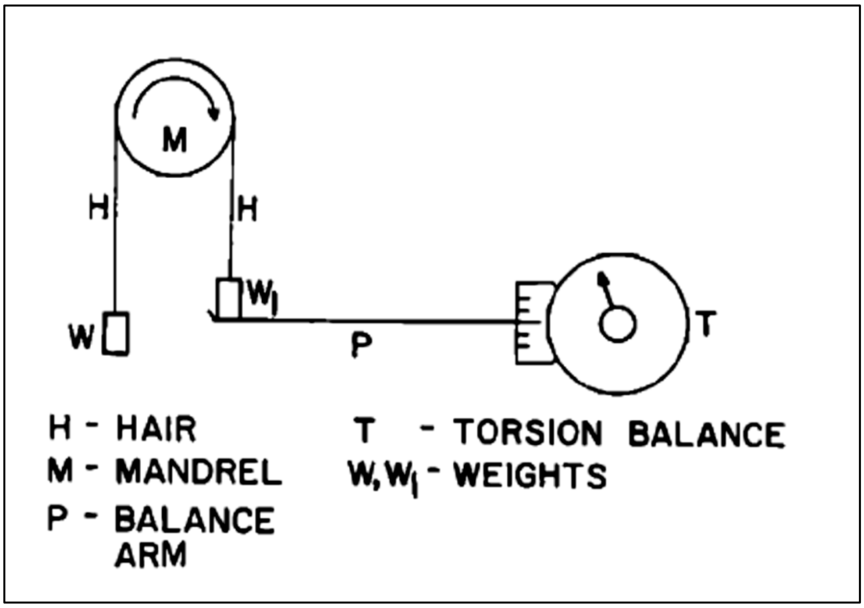
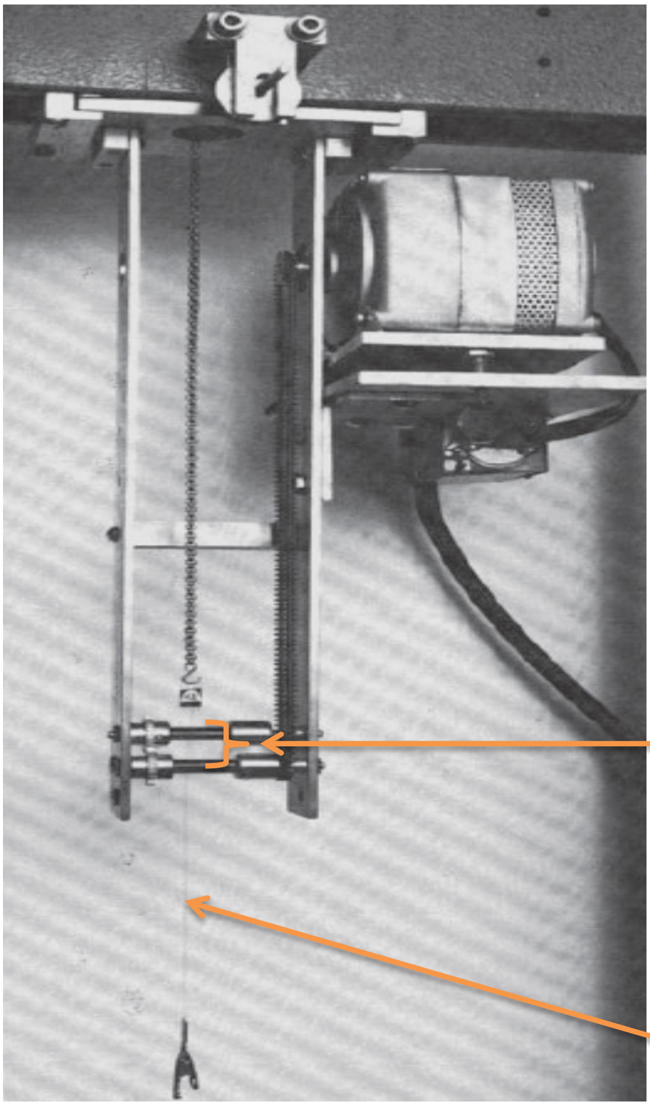


Figure 4.3 Schematic diagram of the instrument used by Schwartz and Knowles [2] .

In the customized Roeder method used by Schwartz and Knowles, the mandrel, M is caused to revolve at uniform viscosity in the direction of the arrow. As M turns, W1 is said to push down on P and the dial of T adjusts so that the balance arm is in the equilibrium position. The frictional force that would be required to maintain a state of equilibrium while the mandrel is moving is W-(W_1 –R). R is the dial reading on the torsion balance T. From this information, the coefficient of friction can then be calculated. Although the mandrel is in motion for the Roeder method, the static friction can be measured by keeping the mandrel stationary. The Capstan method uses a similar mandrel set-up as that in Roeder's studies. Figure 4.4 shows the experimental set-up of the Capstan method by Scott and Robbins.



The study measured the frictional forces of a single hair fibre as it moves over a set of mandrels. Two mandrels were used in the study by Scott and Robbins.

The human hair fibre as it moves over a set of mandrels.

Figure 4.4 An image of the Capstan approach used by Scott and Robbins [1].

The capstan method calculates the frictional coefficient (F.C) ,denoted by the symbol μ, using the following formula:

$$F.C = \frac{1}{\theta}\ln\left(\frac{T_2}{T_1}\right) \qquad \text{(Eqn. 4.2)}$$

where:

Θ = angle (in radians) that the hair is wrapped over the mandrel, relative to itself.

T_1 = tension applied to the lower end of the fibre

T_2 = tension developed at the upper end of the fibre.

Some workers do not use equation 4.2 for hair friction measurements because it has been shown to only be applicable to materials that deform plastically [11, 12]. The method employed in the current study was similar to the capstan approach. A swatch of human hair was wrapped around a rubber mandrel. The rubber mandrel was covered with synthetic hair in order to simulate the movement of hair out of a braid ensemble in the braid detangling process. The usual wrap angle (θ) for capstan methods is 180^0 [2]. The wrap angle used in the current study was also 180^0. See a diagrammatic representation of the experiment set-up in Figure 4.5. With this capstan set-up, both hair ends pointed downward. Figure 4.6 and figure 4.7 show the actual instrumentation used in the current study.

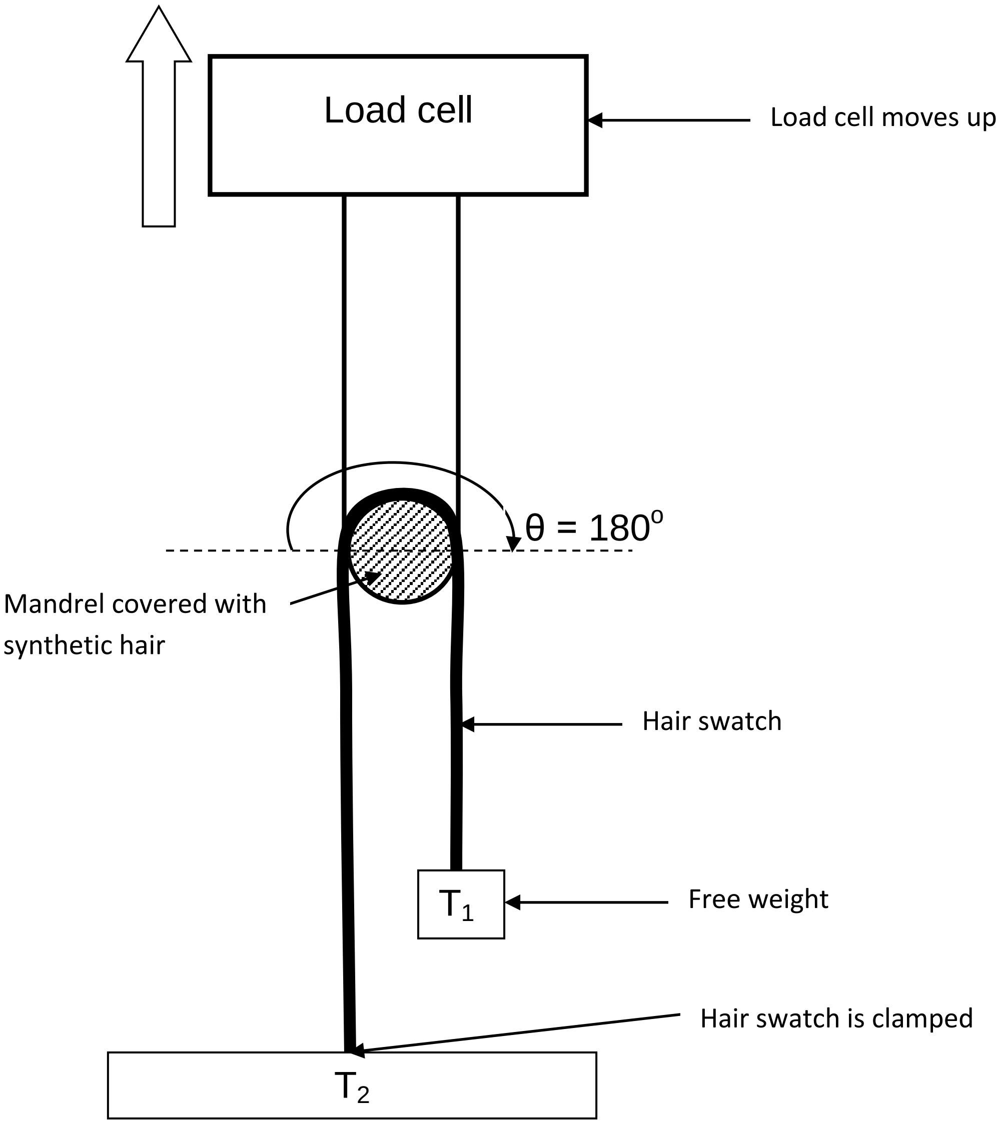
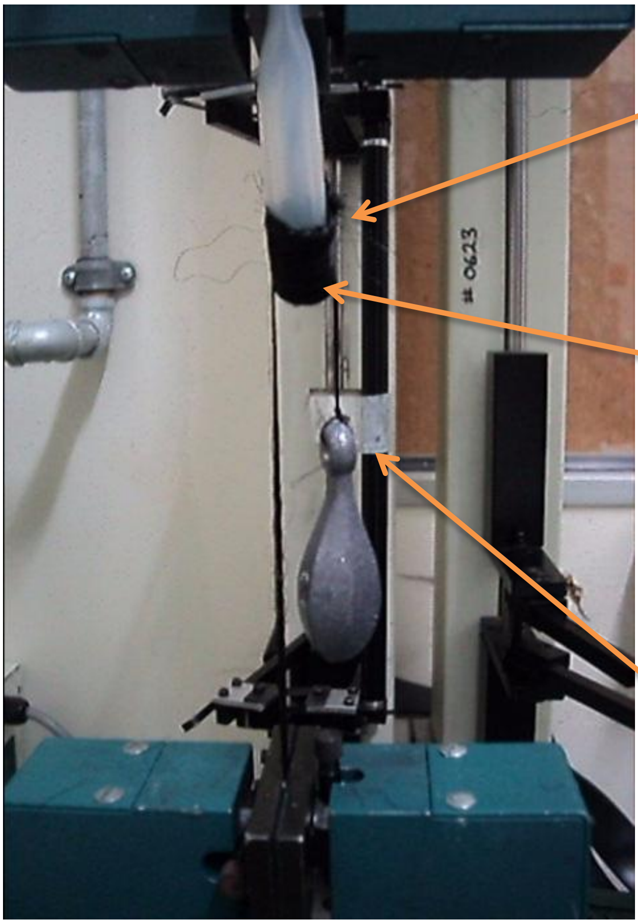


Figure 4.5 Diagrammatic representation of the adapted capstan method

Figure 4.6 A full image of the Hounsfield tensometer H25KT used in the current study for the measurement of friction. The tensometer is housed at Aberdare Cables, Stanford, Port Elizabeth, South Africa.

The hair swatch was clamped on the bottom clamp of the Hounsfield tensometer. The mandrel was clamped on the top clamps, the load cell was connected to the top clamp and the force required to cause the hair swatch to move over the mandrel could be read on a computer screen connected to the Hounsfield tensometer. The hair swatch is weighted at the unclamped end.



T2 (kg) is obtained from the tension developed at the upper end as the hair moves over the mandrel. This tension is recorded by a load cell attached to the extensometer. T2 was calculated using F=m*g.

The mandrel; rubber tube covered with synthetic hair. The mandrel was made by wrapping a known weight of synthetic hair around a rubber tube. The synthetic hair was wrapped once over each uncovered section. The tube was completely covered in the testing region.

T_1, tension at the end of the hair swatch. (T_1=0.0223kg) .The weight is tied by the tip of the hair swatch. The hair moves from root to tip, in the direction of the lowest frictional forces.

Figure 4.7 A close –up of the experimental set-up.

The hair swatch was moved over the synthetic hair-covered mandrel at a constant velocity of 15 mm/min. The force was measured over a 30 mm section. The median of this force was then recorded as the representative frictional force for that particular sample. This median force was then converted to T_2 by dividing the frictional force with the gravitational constant, g. The coefficient of friction was then calculated using equation 4.2 and converting the wrap angle (θ) to radians. All measurements were conducted in an environmentally controlled room; relative humidity = 55 ± 5%, T = 23 ± 1^0C.

4.3.2 Hair samples

Dark brown, chemically treated (relaxed) hair samples were obtained from a 24 year old African female. All the hair samples used were 150 ± 10mm in length. These hair samples were used for all the frictional force studies. The average frictional force of a

10mm section was used for the data collection and data analysis for each of the test formulations.

4.3.2.1 Hair cleansing prior to treatment with test formulation

To prepare the hair for analysis, it was cleaned with a 10% sodium lauryl ether sulphate (SLES). The cleaning method was as follows: a 0.07 ± 0.005g hair swatch was weighed on an analytical balance. The hair swatch was soaked in 10 ml of a 10% (SLES) solution for 30 seconds. The hair swatch was then shaken lightly in the SLES solution for a further 30 seconds to remove any residue on the hair swatch from previous treatments. To remove the SLES solution, the hair swatch was then rinsed under medium flow cold running tap water for 60 seconds. The hair swatch was then left to dry at ambient temperature for 4 hours.

4.3.2.2 Hair sample treatment with test formulation

Each clean hair swatch was placed in a clean petri dish. There were 20 test formulations in total, prepared in accordance to the d-optimal mixture design discussed in chapter 2. The test formulations contained silicone lubricants. The presence of lubricants was anticipated to decrease the frictional forces between synthetic hair and human hair in hair that had been treated with any of the test formulations. Each hair swatch, cleaned as outlined in 4.3.2.1 above, was then treated with 3 ml of the test formulation in a petri dish. Each test formulation was then massaged onto the hair swatch to saturate it for 30 seconds. Each hair swatch was then left to soak in the test formulation for a further 30 seconds and then removed from the petri dish. Excess test formulation was removed from the hair swatch by a single downward stroke of the thumb and index finger. The hair swatches were then oven dried at 50˚C for 10 minutes. After the 10 minutes had elapsed, the hair swatches were cooled to room temperature, wrapped in aluminium foil for transportation to the testing site and then mounted for measurement after removing the foil.

4.4 RESULTS AND DISCUSSION

As mentioned in chapter 2, an experimental design was employed to test the effect on the friction of altering some of the ingredient levels. Four different variables (ingredients) were selected. The effects of these ingredients were varied as shown in table 4.2.

4.4.1 Results

The changes in the coefficient of friction of hair samples that were treated with 20 different test formulations (with some replicates) are shown below in table 4.2. A graphical representation of this data can also be seen in figure 4.8. The friction arising from the contact between the synthetic hair and the human hair was interpreted as the hair lubricity. The most favourable test formulation for this study would be the one that showed the lowest lubricity, .i.e. the lowest coefficient of friction. The data analysis of the results is shown below in section 4.4.2.

Table 4.2 Results of D-optimal mixture design employed to evaluate hair friction

Test Form. (n)	X1 (%)	X2 (%)	X3 (%)	X4 (%)	Median F (N)	T2 (kg)	Coeff of friction, µ
1	0	5	5	59	1.44	0.147	0.600
2	0	10	10	49	1.57	0.160	0.628
3	5.625	2.5	7.5	53.375	1.85	0.189	0.681
4	5	5	0	59	1.48	0.151	0.608
5	7.5	10	5	46.5	1.38	0.141	0.587
6	20.625	2.5	2.5	43.375	1.63	0.166	0.640
7	30	0	0	39	1.41	0.143	0.593
8	10	0	0	59	1.42	0.145	0.596
9	15	10	5	39	1.48	0.151	0.608
10	0	0	10	59	1.57	0.160	0.628
11	15	5	0	49	1.61	0.164	0.635
12	0	10	0	59	1.58	0.161	0.630
13	10	0	0	59	1.53	0.156	0.620
14	20	0	10	39	1.37	0.140	0.586
15	15	0	5	49	1.52	0.155	0.618
16	0	10	0	59	1.71	0.175	0.655
17	0	0	10	59	1.49	0.152	0.612
18	20	0	10	39	1.55	0.158	0.623
19	0	10	10	49	1.46	0.149	0.604
20	25	5	0	39	1.74	0.178	0.661

Where X1 = Cyclopentasiloxane; X2 = PE-12 Dimethicone; X3 = 18-MEA; X4 = Water

The response (coefficient of friction, µ) was calculated using equation 4.2 as follows:

$$F.C = \frac{1}{\theta}\ln\left(\frac{T_2}{T_1}\right) \quad = \frac{1}{3.14}\, ln \frac{T2}{0.0223kg}$$

Lubricity is defined by Wikipedia and the Merriam-Webster dictionary to be the measure of the reduction in the friction of a lubricant [13, 14]. The Unabridged Dictionary defines lubricity to be the "oily smoothness, slipperiness of a surface" [15]. In the experiment, test formulation #5 and #14 showed the lowest coefficients of friction. Figure 4.8 below shows a plot of the coefficients of friction obtained for the different test formulations.

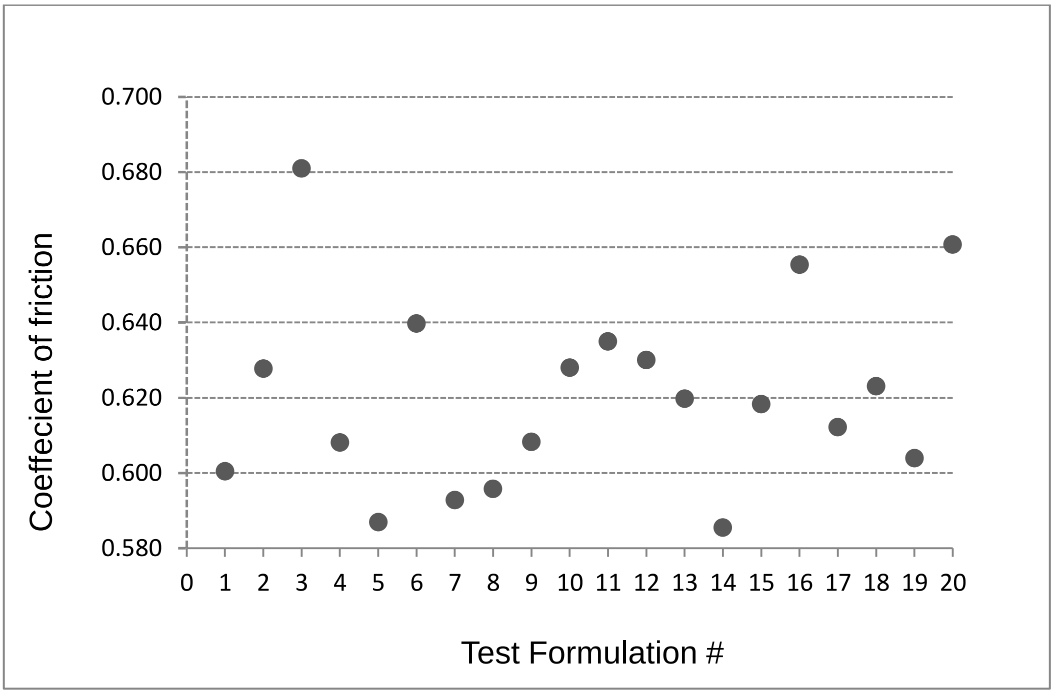


Figure 4.8 A plot of hair friction as indicator of hair lubricity for treated hair swatches.

A decrease in friction is correlated or interpreted to be an increase in the lubricity, or the slipperiness of a surface, thus the test formulation that yielded the lowest coefficient of friction would be most favourable formulation. The best formulation is discussed later in the optimization section of this study.

4.4.2 Data analysis

Multiple regression was used to analyse the measured data. The full proposed model for the regression was:

$$\hat{Y} = b_1X_1 + b_2X_2 + b_3X_3 + b_4 X_4 + b_5X_1X_2 + b_6X_1X_3 + b_7X_1X_4 + b_8X_2X_3 + b_9X_2X_4 + b_{10}X_3X_4$$

(Eqn. 4.3)

Where $\hat{Y}$ is the predicted coefficient of friction.

The experimental model used was based on a mixture design, thus there is no intercept in the full model; no intercept is needed for a regression model that is based on a mixture design. In a mixture experiment, the response variable, in the case μ, is a function of the proportion of each component or independent variable in the mixture or blend [16]. A requirement of a mixture design is that the sum of the proportions (of ingredients varied) in the blend must be equal to the same amount for each blend. The chosen variables made up 69% of the total formulation for each experimental run. A set of constant components made up the remaining 31%. Each blend was 100%, satisfying the guidelines for the analysis of mixture designs. The detailed design is discussed in chapter 2 of the dissertation. The ingredient levels of the four variables under study were constricted as shown in the table 4.3; the chosen levels were based on suggested levels of use for the variables.

Table 4.3 Constraints applied on mixture design

	Ingredient	Minimum %	Maximum%
1	X1 (Cyclopentasiloxane)	0	30
2	X2 (PE-12 Dimethicone)	0	10
3	X3 (18-MEA)	0	10
4	X4 (Water)	q.s	

Using a multiple regression to analyse the data, it was seen that only the independent variables, b_1 to b_4 in equation 4.3, had significant effects on the coefficient of friction. There were no significant changes in friction arising from the interactions between any two of the ingredients that were used in this study. The p-values obtained for all the interactions; b_5 to b_{10} are shown below in table 4.4.

Table 4.4 P-values of insignificant terms in equation 4.3.

Estimated Coeffec.	Interaction	P-value
b10	X3X4	0.779
b8	X2X3	0.757
b7	X1X4	0.256
b6	X1X3	0.307
b9	X2X4	0.145
b5	X1X2	0.478

The null hypothesis related to test the significance of each estimated coefficient was: Ho: $\beta x = 0$. The p-value are all larger than 0.05, thus the null hypotheses had to be accepted. It could not be shown that there were significant interactions between any two of the ingredients used. All the interactions were insignificant. The large p-values, as shown in table 4.4 strongly suggest that the interactions are, in fact, not significant. All four of the ingredients (independent variables, main effects) that were investigated in this study were found to have a significant effect on the frictional forces that arise when human hair moves against synthetic hair. The final model, with only the significant terms included is as follows:

$$\textbf{Final Model: } \hat{Y} = b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 \quad \text{(Eqn. 4.4)}$$

The final model, as shown by the equation above is usually referred to as the main effects table because it only includes the main effects, or the main variables and not any interactions. The p-values of all the four ingredients had low p-values, showing their statistical significance. The definitions and interpretation of p-values and confidence intervals used in this section of the study is the same as that used in chapter 3. Table 4.5 shows a summary of the statistics obtained for the final model.

Table 4.5 Statistics of significant terms (of data in table 4.2)

Regression Statistics	
Multiple R	0.999
R Square	0.998
Adjusted R Square	0.936
Standard Error	0.0270
Observations	20

ANOVA

F	*Significance F*
2639.12	3.70E-21

	Estimated Coeff.	*P-value*	*Lower 95%*	*Upper 95%*
X1 (Cyclopentasiloxane)	0.00882	< 0.0001	0.00758	0.01007
X2 (PEG-12 Dimethicone)	0.00930	< 0.0001	0.00619	0.01242
X3 (18-MEA)	0.00817	< 0.0001	0.00525	0.01110
X4 (Water)	0.00908	< 0.0001	0.00855	0.00960

As can be seen in table 4.5 all the p-values for the main effects were much smaller than 0.05. The true coefficients for each of the main effects can be found between the lower and upper limits, with 95% certainty. The R-squared shows that 99% of the variation in the friction arising when a length of human hair moves along a section of synthetic hair is explained by the model. The 18-MEA was seen to have the lowest estimated coefficient. Thus 18-MEA was the best ingredient in lowering the coefficient of friction.

4.4.3 Model validation

The proposed final model had to be statistically validated. The validity of a model can be shown by a simple plot of standard residuals versus predicted μ in figure 4.9. This plot will show if there are any outliers in the data.

4.4.3.1 ***Test for outliers***

The chosen outlier range for the data was ±2. Standard residuals above or below 2 are evidence of an outlier. Only one outlier was found for the data. This is seen by the data point that is above 2. Furthermore, no distinct pattern was seen for this plot; the standard residuals were randomly distributed around the x-axis, showing that the model fits the data well. This outlier was not removed from the data because removing it did not change the model.

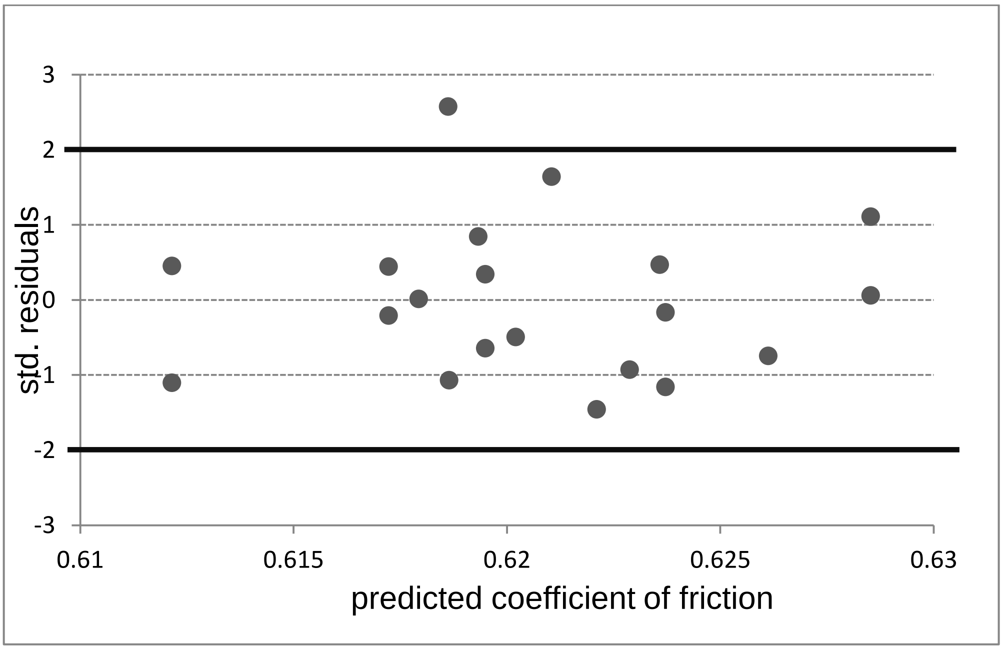
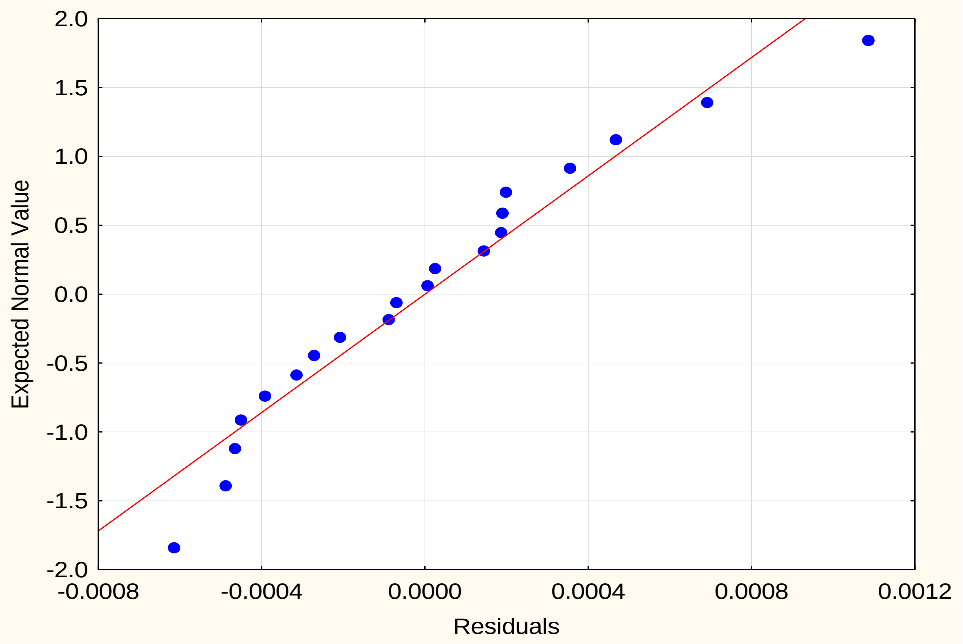


Figure 4.9 Model validation plot showing random distribution of std. residuals around the x-axis, showing that the model fits the data.

4.4.3.2 Test for normality

Using the Lilliefors test for normality, it was accepted that the residuals are normally distributed. The normality plot is shown below in figure 4.10.



4.10 Normal plot of residuals.

4.4.4 Optimization of lubricating formulation

Once the main effects model was validated to be a suitable model for the data, the model could be used to predict the "best" blend. The best blend of ingredients for this study would be one that gives the lowest coefficient of friction. The coefficients of the main effects, as shown in table 4.5, can be substituted in the final model in order to predict the coefficient of friction for any selected blend. Equation 4.5 shows the final model with each of the coefficients of the main effects. Table 4.6 shows the predicted optimum formulation that gives the lowest coefficient of friction.

$$\textbf{Predicted } \mu = \quad 0.00882*X1 + 0.00930*X2 + 0.00817*X3 + 0.00908*X4$$

(Eqn 4.5)

Table 4.6 Optimization of formulation

X1	X2	X3	X4	Predicted μ
30	0	10	29	**0.610**

The optimum mixture with the minimum coefficient of friction, predicts a coefficient of 0.61 ± 0.04.

From the predicted optimized formulation, it was seen that maximum levels of Cyclopentasiloxane (X1) and 18-MEA (X3), no PEG-12 Dimethicone (X2) and the minimum amount of water (X4) would yield the lowest coefficient of friction. This was the best blend. Some of the observed coefficients of friction obtained for the test formulations in table 4.2 show a coefficient of friction that is lower than the predicted optimized formulation. This is thought to be due to experimental error. To confirm the predicted friction coefficient for the optimization, a confirmation experiment was conducted, the results of which are shown in section 4.4.5 below.

4.4.5 Confirmation experiment

In order to verify the optimized formulation, 5 replicate formulations were made up as outlined in chapter 2 with the levels of ingredients set as in table 4.6. Hair swatches were exposed to each of the 5 formulations as outlined in section 4.3.2. The frictional forces of these hair swatches were measured and the coefficient of friction was calculated for each replicate using equation 4.2. The results for this experiment are shown below in table 4.7.

Table 4.7 Results and statistics of confirmation experiment

Sample #	Coefficient of friction
1	0.672
2	0.672
3	0.653
4	0.634
5	0.640
Sample mean	0.654
t	2.776
std. dev.	0.017
n	5
LL	0.632
UL	0.676

The mean coefficient of friction for the 5 replicate samples was found to be 0.654. The true coefficient of friction lies between 0.676 and 0.632 .The predicted optimum coefficient of friction (0.610) lies outside this range. Ideally, the predicted coefficient of friction from the model should be within the confidence interval of the confirmatory experiments. This was not the case in this study. This was thought to be due to the nature of the response surface; the hair shaft. It has been reported that the properties of hair sometimes differ, even for hair samples sourced from the same head and region [1].

4.4.6 Testing the research hypothesis

The research hypothesis for this chapter was as follows:

"Introducing a lubricating formulation between the human hair and the synthetic hair that make up the braid will decrease the friction between the two surfaces."

In order to test this hypothesis, the coefficient of friction for untreated hair was measured. Three replicate untreated hair samples were measured. The average coefficient of friction for these samples was calculated using equation 4.2. The average coefficient of friction for the three replicates was 0.689. The 95% confidence interval for the predicted optimum formulation was calculated using the full data set. The upper limit and lower limits are shown below.

Predicted μ	0.610
95.0% CI Lower limit	0.570
95.0% CI Upper limit	0.649

The average coefficient of friction of the untreated hair swatches (0.689) falls outside this confidence interval, showing that adding a lubricating formulation between the human hair and the synthetic significantly decreased the hair friction. The research hypothesis for this study was accepted.

4.5 SUMMARY AND CONCLUSIVE REMARKS

The objectives of this chapter, as outlined in section 4.2.1, were achieved. A method to measure some of the frictional forces present in the braid detangling environment was developed and used to measure friction and to get an indication of the lubricity of the human hair. The predicted formulation that would yield the lowest friction or highest lubricity was calculated using estimated coefficients that were obtained from a multiple regression. The research hypothesis was accepted; the study found that the frictional forces of all the treated hair swatches were significantly lower than that of untreated hair swatches. This can be translated to mean that treating one’s hair with any of the test formulations would result in easier braid detangling; the proposed formulation was suitable to facilitate the braid detangling process because it minimised some of the frictional forces that would be present within the braid.

4.6 SUGGESTED FUTURE WORK

The estimated coefficients of friction for the chosen lubricants in this experimental design were very similar, showing that their impact on the frictional properties are also similar; a recommendation and improvement to the study would be to study a selection of superior lubricants.

4.7 REFERENCES

1. Clarence R. Robbins. The physical and mechanical behaviour of human hair. 3rd Edition. 1994 ,Springer-Verlag
2. Anthony M. Schwartz, D.C Knowles. Frictional effects in human hair. Presented April 1963, Chicago.
3. Nancy Fair, B.S Gupta. Effects of Chlorine on friction and morphology of human hair. J. Soc Cosmet. Chem. 1982; 33: 229 -242.
4. Carmen La Torre, Bharat Bhushan. Nanotribological effects of silicone type, silicone deposition level and surfactant type on human hair using atomic force microscopy. J. Soc. Cosmet. Chem 2006; 57: 37 -56
5. Steven Breakspear, James R. Smith, Gustavo Luego. Effects of the covalently linked fatty acid 18-Methyleicasonoic acid on the nanotribology of the hair’s outer surface. Journal of structural biology 2005; 149: 235 -242
6. Ali N. Syed. Hair damage: its causes, prevention and cures. Avlon industries Inc., Bedford Park, Illinois.
7. Carmen La Torre, Bharat Bhushan. Investigation of scale effects and directionality dependence on friction and adhesion of human hair using AFM studies and macroscale friction test apparatus. Ultramicroscopy 2006; 106: 720 -734.
8. Nonhlanhla. P Khumalo, Susan Jessop, Freedom Gumedze, Rodney Ehrlich. Determinants of marginal traction alopecia in African girls and women. J Am Dermatol 2008;59:438-9

9. G.V Scott, C. R Robbins. Effects of surfactant solutions on hair fibre friction. J. Soc. Cosmet. Chem 1980;31: 179 -200
10. H. L Roeder. Physics of fibrous materials .Symposium of Dutch Physical Society, Arnhem, The Netherlands, 1950
11. F. P Bowden, D. Taber. The friction and lubrication of solids, Oxford University Press, London, 1950, revised reprint, 1954.
12. Bharat Bhushan, Guohou Wei, Paul Haddad. Friction and wear studies on human hair and skin. Wear 2005; 259: 1021 -1021.
13. http://en.m.wikipedia.org/wiki/Lubricity (accessed January 2012)
14. http://www.merriam-webster.com/dictionary/lubricity (accessed January 2012)
15. http://m.dictionary.com/d/?q=lubricity&o=0&l=dir (accessed 2012)
16. http://www.statsoft.com/textbook/experimental-design/#mixture (accessed January 2012)

CHAPTER 5

INVESTIGATION OF THE TENSILE PROPERTIES OF CHEMICALLY TREATED AFRICAN HAIR.

5.1 INTRODUCTION

5.1.1 Chemical and physical characteristics of human hair

The tensile properties of human hair are an important metric of its strength and its resistance to fracture [1]. The strength of the hair fibre is said to be proportional to its cross sectional area; the thicker the hair fibre, the stronger it is [2]. The tensile strength properties of human hair are attributed to orientation of the helical chains of keratin polypeptides to the longitudinal axis of the hair [3]. The keratin polypeptides are orientated in parallel to the longitudinal axis of the hair shaft. The keratin that makes up the hair is rich in cystine [3]. The disulphide bonds of the cystine protein in the keratin polypeptides are responsible for the mechanical properties of human hair [1]. Figure 5.1 shows the different chemical bonds present in the keratin chains.

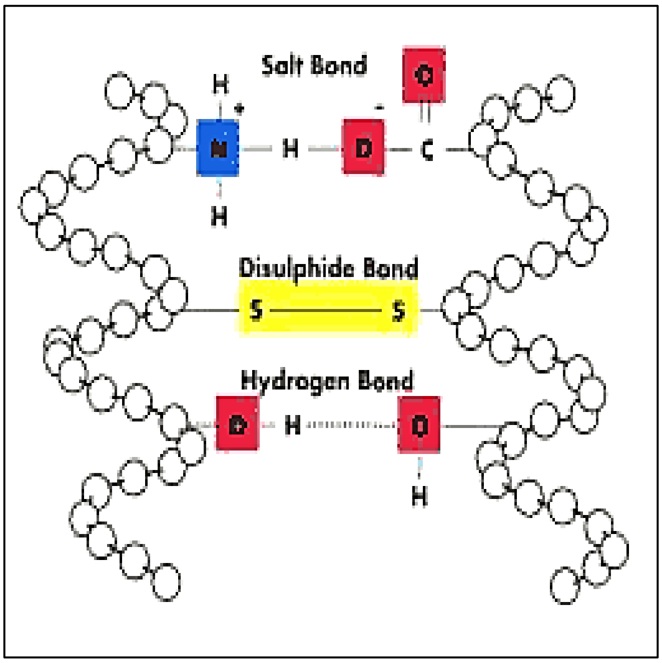


Figure 5.1 Chemical bonds present within the hair shaft.

When a hair strand is stretched, a plot of load versus percentage elongation shows the occurrence of three regions: the pre-yield region, the yield region and the post-yield region (See figure 5.2). The pre-yield region is sometimes referred to as the Hookean region. In this region the hair has the ability to return to its original length when the force exerted to stretch it is released [2]. In the Hookean region the hair is said to show a high resistance to being stretched. This resistance is said to be a result of the hydrogen bonds that are present in the hair. The hydrogen bonds work by stabilizing the alpha-helix of the keratin. The effects of the absence of these hydrogen bonds can be seen when comparing wet hair with dry hair. Wet hair requires less force to stretch it than dry hair. This is because in wet hair, the hydrogen bonds have been disrupted.

In the second region; the yield region, the keratin transitions from being an alpha form to a beta form. In this region, less force is required to stretch the hair, however once stretched into this region, the hair fibre does not return to its original length. The damage inflicted on the hair in this region to cause it to stretch is also said to be more permanent [2]. The chemical bonds that influence this region are primarily hydrogen bonds and salt links.

The last region is the post-yield region. In this region the beta configuration of the keratin structure resists stretching and more force is required to extend the hair until it reaches breaking point. The dominant bonds in this region are covalent bonds in the protein. When a stress versus strain curve of the hair is plotted, as in figure 5.2 below, the yield region is observed at about 5% strain and the post yield starts at 15% strain [4]. When the hair fibre is stretched to about 30% of its initial length, cracks are said to appear on the cuticle and eventually the cuticle will start to separate from the cortex.

Tensile strength is defined as the measure of the force required to pull something to a point where it breaks [5].

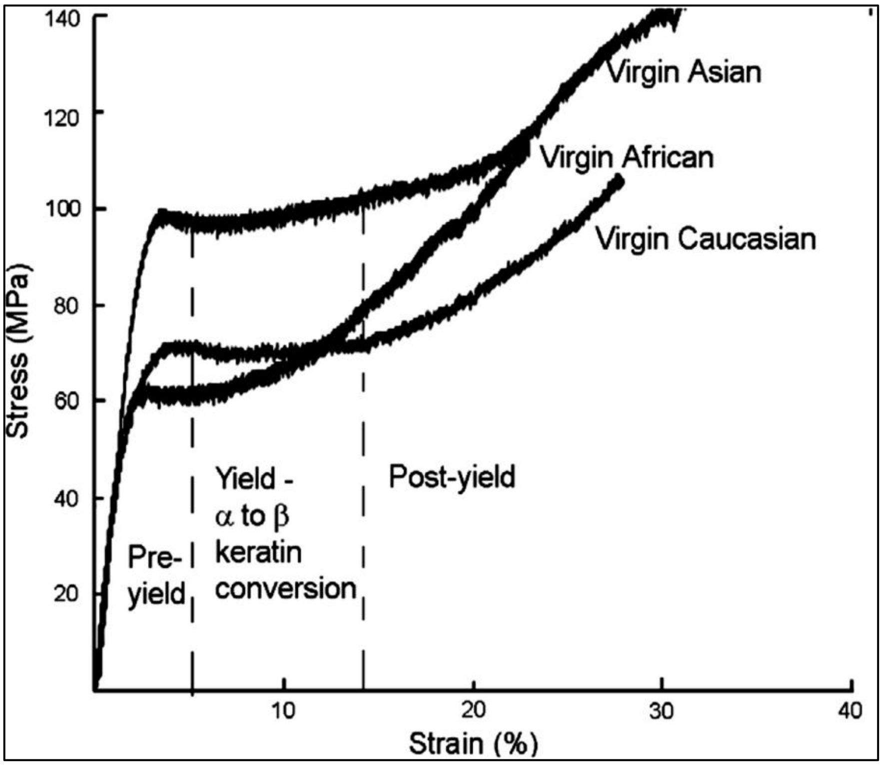


Figure 5.2 Stress-strain curves for Caucasian, Asian and African hair [1].

Young's elastic modulus (E) is the measure of how much force is needed to stretch or compress a substance. Young's modulus shows a relationship between stress and strain. Stress is the measure of a force in a certain area. Strain is the amount that the substance is deformed [5]. Young's elastic modulus is better illustrated by the following equation:

$$E = \frac{stress}{strain} = \frac{F/_{A}}{\Delta L/_{L}} \qquad \text{(Eqn.5.1)}$$

Where E = Young's modulus (modulus of elasticity)
F = force exerted on the object under tension
A = cross-sectional area
ΔL = the amount by which the length of the object changes
L = original length of the object

The units for Young's modulus are Pascal (Pa).

5.1.2 Characteristics of African hair

When hair from different ethnicities is evaluated, African hair has been seen to have unique characteristics that influence its manageability [6, 7, and 8].

- Chemical composition: similar, consists of proteins and amino acids.
- Diameter: irregular, African hair has an elliptical shape.
- Physical shape of fibre: has small twist and inconsistency in hair cuticle diameter along the length of the hair shaft.
- Combability: difficult to comb because of its curly configuration
- Mechanical properties: African hair is more fragile than Asian or Caucasian hair [6][8]. This was observed to be the case by Kamath and Weigmann even for virgin African hair [9]. This can also be seen in figure 5.2 where the yield strength of African hair is 58 MPa, whilst that of Asian and Caucasian is 100 MPa and 67 MPa respectively.
- Density: African hair is less dense and is said to grow more slowly than Caucasian or Asian hair.
- Hair moisture: African hair has a lower moisture content than Caucasian and Asian hair.

The tensile strength of African hair has been reported to be lower than that of Caucasian or Asian hair [6]. This difference in the tensile strength of African hair is hypothesized by several workers to be the result of the physical shape and the moisture content of the African hair fibre [9]. In a study by Kamath and Hornby, it was seen that increasing the moisture content of African hair by treating it with humectants, showed a reduction in hair breakage [9]. Table 5.1 shows the differences in the cross-sectional properties of African, Asian and Caucasian hair.

Table 5.1 Differences and similarities in cross-sectional dimensions of hair according to ethnicity [9, 10].

Ethnicity	Shape	Max. D1(μm)	Min. D2 (μm)	No. of scales	Cuticle Scale Thickness(μm)
African	Oval-flat	89	44	6 - 7	0.3 - 0.5
Asian	Nearly	92	71	5 - 6	0.3 - 0.5
Caucasian	Nearly	74	47	6 - 7	0.3 - 0.5

The differences in the hair structure and manageability of African hair compared to hair of other ethnicities often leads to excessive force and measures being applied to style African hair. In a study by Khumalo et al, Traction Alopecia (TA) was associated with hairstyles that are prevalent in the hair grooming practices of African females [10]. The application of traction, as in the case of hair braiding, on chemically treated hair was found to be amongst the highest contributors to styling TA [10]. Table 5.2 shows the calculated mechanical properties of hair from different ethnicities.

Table 5.2 Mechanical properties of hair samples of different ethnic origin.

	African	Asian	Caucasian
Elastic modulus (GPa)	2.5	4.7	3.3
Yield strength (MPa)	58	100	67
Breaking strength (MPa)	101	139	117
Strain at failure (%)	20	32	35

The elastic modulus is calculated by fitting a line to the linear portion of the stress-strain curve as seen in figure 5.2 and finding its slope. The yield strength is taken as the highest point of this linear region. The strain and stress at the point where the fibre fails are taken as the strain to break and the breaking strength respectively [1].

5.1.3 Grooming African hair: Braiding

Braiding is one of the common grooming practices among African females and some African males. In hair braiding practices, manufactured hair is interwoven with the wearer’s natural hair to form a braid. The wearer’s hair can be relaxed or unrelaxed (natural). These braids are generally worn for 4-8 weeks and then removed. The removal of braids involves the use of a fine toothed-comb and safety pins. The pointed ends of such tools are put in the braid and a force is exerted to pull apart the braid. Once the braids are detangled, more efforts are spent in detangling or combing out knots in the hair to prepare it for the next hairstyle. The knots in the hair are caused by entanglement of dead hair (hair that has been detached from the scalp) that has been shed when the hair was worn in the braided hairstyle with the intact hairs. Human beings shed approximately 100 hairs per day. During the time the braids are worn the hair would be washed every second week and the braids maintained using hair pomades and hairspray. The hair is, however, more brittle because it is not moisturised as well as it would have been if it was not in braids. If excessive force is applied to detangle these knots, some hair damage and ultimately hair breakage is inevitable. Repeated combing and picking of the hair assembly is said to be equivalent to subjecting the hair fibres to cyclic tensile loading or fatigue [9]. The entanglement of hair fibres is minimized by lowering adhesive forces; figure 5.3 shows the hair-to-hair interactions of entangled hair [11].

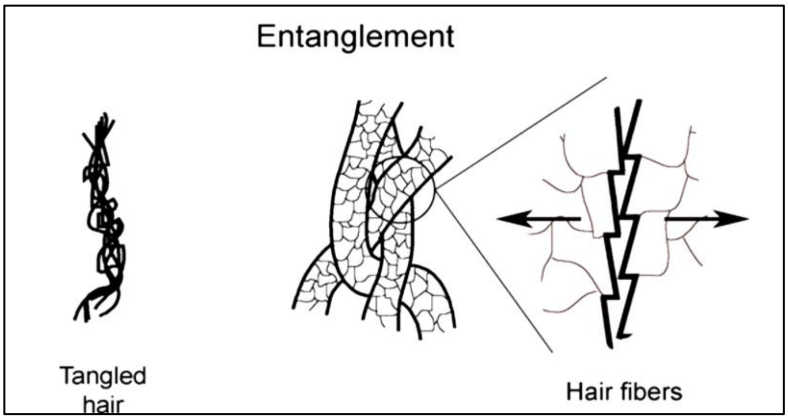


Figure 5.3 Schematic illustration of macro, micro and nanoscale characterization of human hair entanglement.

5.1.4 Hair strengthening actives

In the broader study of this dissertation (see chapter 1 and 2), the formulation proposed contains hydrolysed protein actives that are said to improve the tensile properties of hair. The two actives that were used in this study to evaluate differences in tensile properties were hydrolysed Elastin (HYDROLASTAN, Pentapharm) and the hydrolysed silk protein of Sericin (SETAKOL®, Pentapharm). These actives are widely used in the skin and hair care industry. HYDROLASTAN and SETAKOL® are reported by their manufacturer to show an ability to improve tensile properties of human hair [12 -14].

5.1.4.1 Hydrolysed Elastin

Elastin is an insoluble protein found in connective tissue. It is elastic and allows body tissues to resume their shape after being stretched or contracted [15]. In the hair care industry, elastin is used for moisturising and conditioning. HYDROLASTAN is mainly composed of the low molecular weight elastin peptide. It also contains small quantities of elastin peptides of a higher molecular weight and some naturally occurring collagen hydrolysates [12]. HYDROLASTAN is said to have an affinity for hair and skin and to be a protective protein.

5.1.4.2 Hydrolysed Sericin

SETAKOL® is a hydrolysate of Sericin, a silk protein. Sericin is said to have an affinity for proteins, binding strongly to the keratin of skin and hair [12]. Once the SETAKOL is bound onto hair or skin, it is said to form a protective film. Both these actives are said to improve the tensile strength of hair in vitro [12].

5.1.5 Research Hypothesis

This section of the study proposes that*:*

"Treating human hair with a formulation containing hair strengthening actives will increase the hair's tensile strength".

An increase in the tensile strength would then be translated into an anticipated decrease in hair breakage during the braid detangling process.

5.1.6 Aim of study

The aim of this part of the study was to evaluate the effects of hydrolysed proteins on tensile strength properties of chemically treated African hair. This was done to propose the inclusion of these actives in a formulation that would constitute an efficient formulation to facilitate the detangling of braids from hair. Increasing the tensile strength of the hair fibres would translate to a reduction in hair damage and reduction in hair breakage during the braid detangling processes.

5.2 EXPERIMENTAL

In this part of the study, the yield strength of a group of hair fibres (hair swatch) was measured. The fibres had a history of chemical treatment (relaxed hair). The yield strength was measured for treated hair and untreated hair using a Hounsfield tensometer. The treated hair was treated with selected test formulations from the experimental design discussed in chapter 4 (see table 4.2). Test formulations 1 to 7 were selected in order to evaluate the effects of the hair strengthening actives and those of the other variables in the formulations. From these measurements, the mechanical properties of each hair swatch could be obtained. The tensile properties of three samples settings were measured in order to establish the best environment for braid detangling; untreated, treated dry and treated wet. For the treated wet hair versus treated dry hair studies, test formulation #8 from the experimental design in chapter 2, table 2.11 was used.

5.2.1 Method and materials

The usual procedure to measure the tensile properties of human hair is by load elongation methods [5] [15]. In these methods, a fibre of known length is stretched at a fixed rate. For this study, the same principle was applied. For the tensile strength measurements, hair swatches were mounted on a jig and pulled at a fixed rate until they broke. The load versus the extension was recorded on a computer connected to the tensometer. From this data, the stress and strain curve could be obtained. All measurements were conducted in an environmentally controlled room; Relative humidity = 55 ± 5%, T = 23 ± 1^0C.

5.2.1.1 Hair samples

Dark brown, chemically treated (relaxed) hair samples were obtained from a 24 year old African female. All the hair samples used were 150±10mm in length. These hair samples were used for both the untreated and treated hair tensile studies. A different set of hair was used for the treated dry versus treated wet hair measurements.

5.2.1.2 Hair cleansing prior to treatment with test formulation

To prepare the hair for analysis, it was cleaned with a 10% sodium lauryl ether sulphate (SLES) solution. The cleaning procedure was as follows: a 0.07 ± 0.005 g hair swatch was weighed on an analytical balance. The hair swatch was soaked in 10 ml of a 10% (SLES) solution for 30 seconds. The hair swatch was then shaken lightly in the SLES solution for a further 30 seconds to remove any residue on the hair swatch from previous treatments. To remove the SLES solution, the hair swatch was then rinsed under medium flow cold running tap water for 60 seconds. The hair swatch was then left to dry at ambient temperature for 4 hours.

5.2.1.3 Hair sample treatment with test formulation

The clean hair swatch was placed in a clean petri dish. The swatch was then treated with 3 ml of the test formulation containing the hydrolysed protein actives; the formulation was massaged onto the hair to saturate it for 30 seconds. The hair swatch was left to soak in the test formulation for a further 30 seconds then removed. Excess test formulation was removed from the hair by a single downward stroke of thumb and index finger. The hair swatch was then oven dried at 50˚C for 10 minutes. After the 10 minutes had elapsed, the hair swatch was allowed to cool down and then wrapped in aluminium foil. For wet tensile measurements, the hair swatch was treated the same way but without being oven-dried. Table 5.3 shows a typical formulation used in the study.

Table 5.3 A typical formulation used in the current study.

Ingredient	% in formulation
Cyclopentasiloxane	20
PEG-12 Dimethicone	5
18-Methyl Eicasenoic Acid	5
Isopropyl Palmitate	3
SETAKOL®	5
HYDROLASTAN	5
Hydroxyethyl cellulose	0.2
Cetyl trimethylammonium chloride	2.50
Lanolin	0.15
Oleth -5	10
Olive oil	5
Water	Qs

5.2.2 Experimental set-up

One of the main challenges encountered in the study was the availability of suitable instruments to measure the chosen response variables of the study. For the tensile strength measurement, a tensile tester in the university's polymer department did not provide enough grip for the hair samples and the hair fibres slipped out of the grips before reliable measurements could be taken. Another tensile tester, housed at the neighbouring Chemical Research Science Institute (CSIR) was only suitable for longer hair strands, and could only measure single fibres. The instrument used in this study was a Hounsfield tensometer housed at Aberdare Cables, a cable manufacturing company in Stanford Road, Port Elizabeth.

The hair was clamped into the top and bottom grips of the Hounsfield tensometer. The human hair samples were then stretched at a constant rate of 15 mm/min until all the hair strands were broken. As the hair swatch was stretched, an increase in the load was observed. The gauge length of the tensometer was set to 100 mm. The Young's modulus was obtained by taking the slope of the linear region of the stress-strain curve (see figure 5.5). The average maximum force reached before all the hair strands broke was recorded. This recorded maximum force was converted to the yield strength by dividing the force with the cross-sectional area of the hair swatch. The yield strength was used as an indication of the tensile strength of the hair fibre for a particular sample. Because of the multiplicity of strands in the swatch, different breaking points were observed for the hair swatch until all the hairs in the swatch broke (failure point) .The experimental set-up is shown in figure 5.4.

Figure 5.4 The Hounsfield H25KT tensometer used for tensile measurements. Housed at Aberdare Cables, Port Elizabeth, South Africa.

5.3 RESULTS AND DISCUSSION

5.3.1 Results: Dry treated hair

The tensile strength of treated hair swatches was measured. The test formulations used were prepared according to the experimental design in chapter 4. Test formulations 1 to 7 from the design were selected for this study. A graphical representation of the results is shown below in figure 5.5. The stress-strain curves were not smooth curves but had a jagged configuration, this was attributed to the multiplicity of hair strands in the hair swatch; it is likely that there were variations in the mechanical properties of individual hair fibres within the swatch.

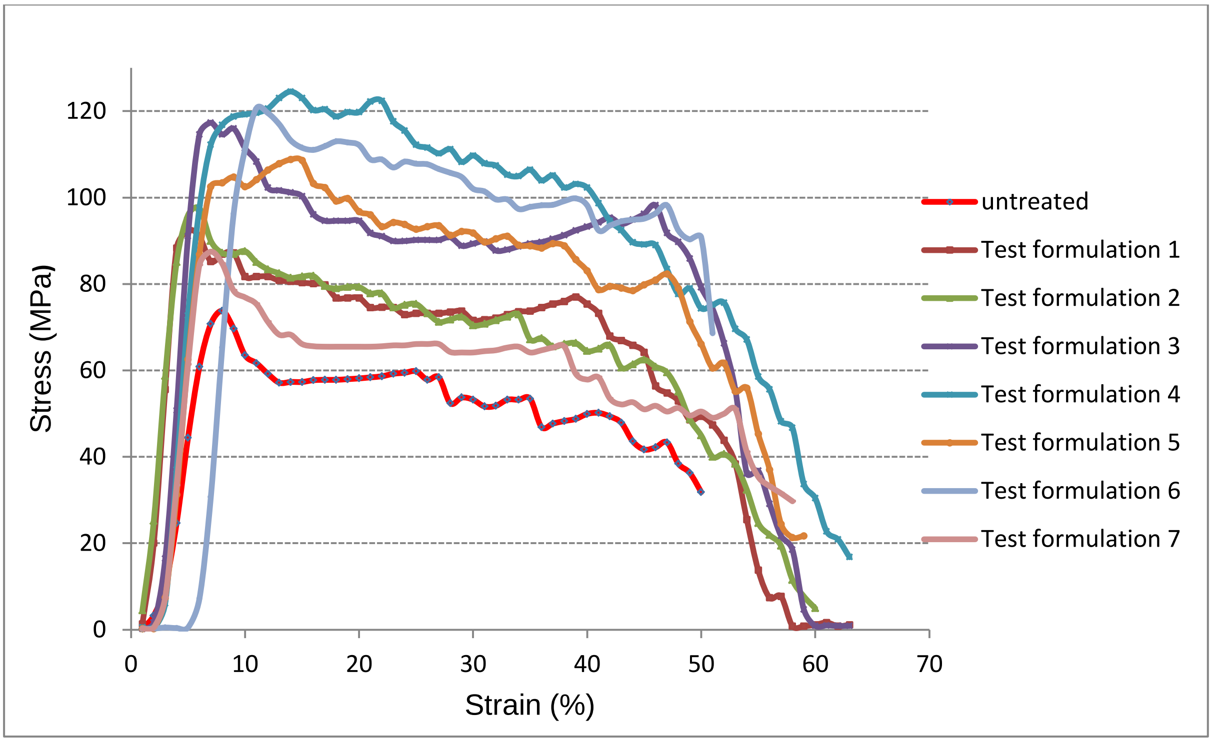


Figure 5.5 Stress-strain curve of treated hair versus untreated hair (blank)

The differences in the maximum load and thus the yield strength of the different runs (1-7) were thought to be due to the interactions arising from the variations of the other active ingredients in the test formulations (see full experimental design in table 2.11). These variations were tested for significance using a one way ANOVA. The statistics for the ANOVA are shown below in table 5.4.

Table 5.4 A summary of ANOVA for treated hair swatches.

Groups	Average load (N)
Test formulation 1	59.57
Test formulation 2	63.03
Test formulation 3	76.05
Test formulation 4	88.41
Test formulation 5	78.19
Test formulation 6	88.61
Test formulation 7	57.31

F-statistic	P-value	F critical
11.61575764	5.02313E-12	2.120694762

The variations in the tensile properties of treated hair swatches were not due to experimental error. The ANOVA gave a p-value that was < 0.00001, implying that there are significant differences in the true average load that treated hair swatches can withstand. The null hypothesis related to the reported p-value stated that there were no significant differences in the tensile properties of hair swatches treated with test formulations containing the same level of hair strengthening actives and emulsifiers but different levels of water and lubricating oils. The null hypothesis was rejected. The finding was that there was evidence of significant differences in the tensile properties. The differences observed for the treated hair were attributed to the variations of some of the ingredients in the test formulations. The levels of hair strengthening actives were constant for all the test formulations. Table 5.5 shows the composition of the test formulations and the variables that could have given rise to the differences in tensile properties. The constant variables, along with the hair strengthening actives accounted for 31% of the formulation's composition.

Table 5.5 Effects of variable components on hair tensile properties

Test form. (T)	Cyclopentasiloxane X1	PEG-12 Dimethicone X2	18-MEA X3	Water X4	Yield Strength (MPa)	Avg. Load (N)
1	0.00	5.00	5.00	59.00	92.43	59.57
2	0.00	10.00	10.00	49.00	97.57	63.03
3	5.63	2.50	7.50	53.38	117.33	76.05
4	5.00	5.00	0.00	59.00	124.53	88.41
5	7.50	10.00	5.00	46.50	108.82	78.19
6	20.63	2.50	2.50	43.38	120.66	88.61
7	30.00	0.00	0.00	39.00	87.49	57.31

5.3.2 Dry treated hair versus dry untreated hair

The calculated results of the mechanical properties of all the hair swatches, treated and untreated, are shown below in table 5.6. The yield strength was calculated by taking the force at yield point and dividing it with the cross-sectional area of the hair swatch. The cross-sectional area of the hair swatch was obtained by calculating the average cross-section of individual hair fibres and then multiplying that cross-sectional area with the number of hair strands in the swatch. Each swatch was comprised of approximately 80 hair strands. The average diameter of the individual hair strands was 45 µm.

Table 5.6 Mechanical properties of untreated (dry) and treated hair (dry).

Specimen	Yield strength (MPa)	Strain at Yield strength (%)	Strain at Failure (%)	Young's Modulus (GPa)
untreated	73.78	9	50	1.22
Test form. 1	92.43	5	58	2.50
Test form. 2	97.57	6	60	2.01
Test form. 3	117.33	7	60	2.40
Test form. 4	124.53	14	63	2.04
Test form. 5	108.82	14	59	1.91
Test form. 6	120.66	11	51	1.33
Test form. 7	87.49	7	58	1.73

The yield strength for all the treated swatches was seen to be higher than that of untreated hair. These differences in yield strength were found to be statistically significant. A series of tests was employed on the data. The null hypothesis for the t-tests was: Ho: $\mu_{\text{untreated}} = \mu_{T1} = \mu_{T2} = \ldots \mu_{T7}$. All p-values obtained were <0.05, as can be seen in the second column of table 5.7. The tensile properties of untreated hair differed significantly from that of all the treated hair. Table 5.7 shows a summary of the p-values obtained for the t-tests of treated hair versus untreated hair as well as the p-values between the different test formulations (T). It can be seen that in most cases the test formulations differed significantly from each other (p values <0.05).

Table 5.7 Summary of p-values of t-tests between treated hair and untreated hair as well as between runs 1 -7.

	Untreated	**T1**	**T2**	**T3**	**T4**	**T5**	**T6**	**T7**
Untreated		0.03369	0.0010	<0.000001	<0.000001	<0.000001	<0.000001	0.0259
T 1	0.03369		**0.4630495**	0.004208	<0.000001	0.000402	<0.000001	**0.6042**
T 2	0.0010	**0.46305**		0.017078	<0.000001	0.001833	1.32E-05	**0.1411**
T 3	<0.000001	0.004208	0.0170777		0.04930	**0.71103**	**0.05918**	0.000364
T 4	<0.000001	<0.000001	<0.000001	0.049295		**0.07972**	**0.97529**	<0.000001
T 5	<0.000001	0.000402	0.0018329	**0.71103**	**0.079716**		0.08759	<0.000001
T 6	<0.000001	<0.000001	1.32E-05	**0.05918**	**0.97529**	0.08759		<0.000001
T 7	0.0259	**0.604157**	**0.141081**	0.000364	<0.000001	<0.000001	<0.000001	

The Table 5.8 shows the statistics obtained for the treated hair swatches. The 95% confidence interval lower and upper limits were found to be 96.23 to 117.23 respectively. The yield strength for the untreated hair fell outside this confidence interval, showing that the true mean yield strength of all the treated hair was significantly different from the true mean yield strength of untreated hair. The differences seen in table 5.6 above were not due to experimental error.

Table 5.8 Statistics for treated hair

Sample mean	106.97
t	1.943
N	7
std. dev.	14.64
Lower Limit	96.23
Upper Limit	117.72

Treating hair swatches with test formulations containing hydrolysed protein actives was seen to show a significant increase to the hair's yield strength. The confidence intervals in table 5.8 show that treating the hair with any test formulation containing hydrolysed protein actives before attempting braid detangling would be more beneficial to the hair than detangling the braids without using a formulation containing hydrolysed proteins.

The calculated tensile properties for African hair in this study varied from those in the literature, as can be seen when comparing table 5.2 with table 5.6. This was thought to be due to the multiplicity of hair strands in the current study. The data in table 5.2 is for single fibres.

5.3.3 Wet hair versus dry hair

The tensile strength of wet hair was compared to that of dry hair. This was done in order to ascertain the best conditions to comb out the knots left behind after braid detangling is achieved. This would also give an indication of how a formulation prescribed to facilitate the braid detangling process would affect the tensile strength properties of the hair. Wet hair sample measurements were measured 10 minutes after the hair swatch had been treated with the test formulation without any oven drying. The hair was still wet. For dry treated samples, the hair swatch was oven-dried at 50 ^{0}C for 10 minutes. One sample was measured for each condition. The hair samples used for the comparisons of wet versus dry hair properties was different from that used in section 5.3.1. The hair used was sampled ~ 10 months before the other hair used in this study. This hair was thus deemed to be stronger, having been exposed to less chemical treatment than the other hair samples used.

The hair swatches were treated with test formulation # 8 from the experimental design. The results for the tensile properties measurements are shown in the stress-strain curve in figure 5.6.

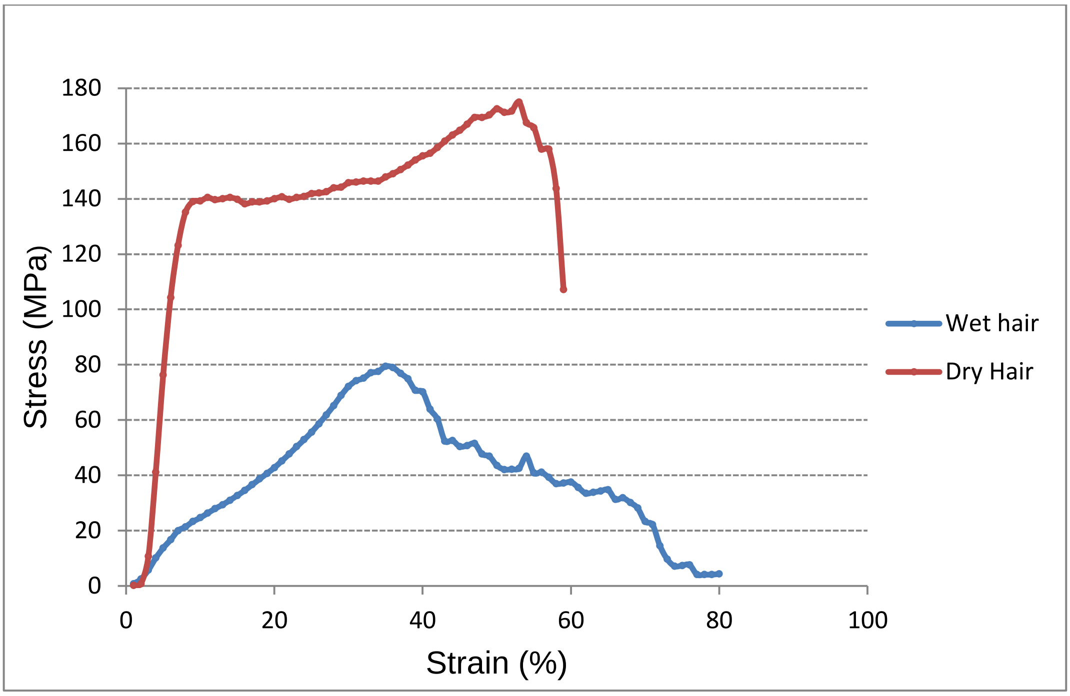


Figure 5.6 The stress-strain curves obtained for the study of dry treated hair versus wet treated hair.

The tensile properties of wet hair are generally different from dry tensile properties. [1] The stress-strain curve obtained in this study as seen in figure 5.6 correlated with scientific literature of soaked hair[1]. The strain to failure of wet hair is greater than that of dry hair, but the yield strength was lower for wet hair. The calculated results are shown below in table 5.9.

Table 5.9 Mechanical properties of wet hair and dry hair.

	Yield strength (MPa)	Strain at Yield strength (%)	Strain at Failure (%)	Young's Modulus (GPa)
Dry	139	9	59	13.9
Wet	77.56	34	80	7.76

The differences in the mechanical properties of wet versus dry hair are attributed to the diametrical swelling of the hair fibre in water. Human hair is said to show 14 -16

% diametrical swelling in water [1, 15]. Literature reports that the mechanical properties of wet hair are affected by chemical interactions within the hair structure. In the wet state, some of the groups that are involved in Coulombic interactions in the α-helix of the keratin interact with water [1]. This interaction breaks some of the Coulumbic bonding, resulting in lower yield strength and a lower wet Young's modulus [1, 15].

A t-test was done to validate the significance of the observed differences in the stress that wet hair could withstand versus the stress that dry hair could withstand. The stress was calculated using the same diameter (45µm) for both samples. The results for the t-test are shown below in table 5.10.

Table 5.10 Significance test result for wet hair versus dry hair.

Ho: $\mu_{wet} = \mu_{dry}$	*Stress (wet)*	*Stress (dry)*
Sample mean	39.27	138.17
Hypothesized Mean Difference	0	
P-value	<0.000001	

The null hypothesis (Ho: $\mu_{wet} = \mu_{dry}$) was rejected, p-value <0.00001. The t-test showed that there is strong evidence that the stress of wet hair differs significantly from the stress of dry hair.

5.4 SUMMARY AND CONCLUSIONS

The tensile strength properties of hair swatches were measured successfully and the yield strength determined. The failure point was also obtained. Although the individual hair fibres broke at different loads, a clear point could be seen where all the fibres in the swatch were broken. Treating the hair fibres with formulations containing hair strengthening actives showed a significant increase in the yield strength of the tested hair swatches. The incorporation of these actives would be beneficial for the hair and would contribute to minimizing hair loss that would be incurred in the braid detangling process.

The tensile strength of dry treated hair was found to be significantly higher than that of wet treated hair. This finding gives the indication that the best tensile environment for braid detangling is when the hair is dry.

5.5 SUGGESTED FUTURE WORK

- Use a full experimental design to evaluate the effects all ingredients present in the proposed formulation on the mechanical properties of hair.
- Compare the tensile strength of treated hair swatches with that of treated individual hair fibres.

5.6 REFERENCES

1. Indira P. Seshadri, Bharat Bhushan. Effect of ethnicity and treatments on in situ tensile response and morphological changes of human hair characterized by atomic force microscopy. Acta Materialia 56 (2008) 3585-3597.

2. Chris Gummer. Society of Cosmetic Chemists Distance Learning. Module 1, Unit 12.

3. John Woodruff. Improving the strength of hair. Published Cosmetics & Toiletries Magazine; 2002.

4. Indira P. Seshadri, Bharat Bhushan. In situ deformation characterization of human hair with atomic force microscopy. Acta Materialia 56 (2008) 741-781.

5. Richard Beyak, C.F. Meyer and G.S. Kass. Elasticity and tensile properties of human hair. I. Single fiber method. J Soc Cosmet Chem 1969; 20:615-626.

6. A. Franbourg, P. Hallegot, F. Baltenneck, C. Toutain, and F. Leroy. Current research on ethnic hair. J Am Acad Dermatol 2003; 48: S115-9.

7. Victoria L. Holloway. Ethnic cosmetic products. Dermatol Clin 2003;21 :743-749

8. Y.K. Kamath, Hornby Weigmann. Mechanical and fractographic behaviour of Negroid hair. J Soc Cosmet Chem 1984; 35:21-43.

9. Y.K. Kamath, Hornby Weigmann. Effect of chemistry and humectant treatments on the mechanical and fractographic behaviour of Negroid hair. J Soc Cosmet Chem 1985; 36:39-52.

10. Nonhlanhla P. Khumalo, Susan Jessop,Freedom Gumedze, and Rodney Ehrlich. Determinants of marginal traction alopecia in African girls and women. J Am Acad Dermatol 2008; 59: 432-8.

11. Bharat Bhusha. Nanoscale characterization of human hair and hair conditioners. Progress in material science 2008:53: 585 -710.

12. http://www.centerchem.com (05/12/11)

13. Marek Gacko. Elastin:structure ,properties and metabolism. Cellular and molecular biology letters. 2000;5: 327-348.

14. http://en.wikipedia.org/wiki/Elastin (05/12/11)

15. Clarence R. Robbins .Chemical and physical behaviour of human hair. 3rd Edition. Springer-Verlag.

CHAPTER 6

6.1 CONCLUSIONS

The main objective of this study was achieved. By carefully looking at the factors that contribute to the ease of braid detangling a cosmetic formulation was developed and optimized. The formulation was developed by matching the desired attributes of an efficient braid detangling formulation to cosmetic ingredients that could provide that desired attribute.

The main objectives of the proposed formulation were to

1. lubricate the hair in order decrease braid detangling forces,

2. lubricate the hair in order to decrease the frictional forces within the braid and to provide “slip” and

3. to improve the tensile properties of the hair in order to minimise breakage and/or damage that would be incurred during the braid detangling process.

1. The first objective of the study was addressed in chapter 3. The research hypothesis for this objective was that:

“Using a tool, typical to tools used in conventional braid detangling processes and those disclosed in patents of braid detangling tools, alongside a formulation would decrease the strain or effort [force] of detangling braids by decreasing the frictional forces in the braid.”

An attempt was made to decrease the braid detangling forces by increasing the lubricity of the hair within the braid by deposition of lubricants onto the hair shaft. This hypothesis was rejected. The method used in the study did not show any significant differences in the braid detangling forces of braids that had been treated with the lubricating test formulations when compared to the braid detangling forces of braids that had not been treated with any formulation. A secondary hypothesis was formulated in this section of the study. The secondary hypothesis stated that:

“**There are significant differences in the braid detangling forces of different sections along the braid.”**

The secondary hypothesis was accepted. The study found that different braid sections had significantly different detangling forces. The braid was demarcated into three sections; section 1 was the section towards the tip of the braid. Section 2 was the middle of the braid and section 3 was the section closest to the root of the hair. These differences are attributed to the physical make-up of the braid. Section 2 was seen to have the largest braid detangling forces; this was attributed to the severe knotting observed in this section. This observation was consistent for all the braided samples.

2. The second objective was studied in chapter 4. The research hypothesis for this objective was as follows:

“Introducing a lubricating formulation between the human hair and synthetic hair that make up the braid will decrease the frictional forces between the two surfaces.”

A decrease in the frictional forces between the human hair and the synthetic hair was interpreted to improve the “slip” between the two surfaces, thereby allowing for easier braid detangling. This hypothesis was accepted. The study found that treating the hair with any of the test formulations would significantly decrease the frictional forces between the human hair and the synthetic hair. The frictional forces were measured by sliding a swatch over a mandrel covered with synthetic hair. This set-up simulated the braid detangling environment where braids are removed by easing the human hair out of the synthetic hair. The frictional forces of treated hair were significantly different from those of untreated hair showing that applying the proposed formulation for braid detangling would be beneficial in allowing easier braid detangling. The study was conducted in vitro, to evaluate the validity of these findings. An in vivo study is recommended where other factors, such as how the braid is plaited are taken into account.

3. The third objective of the study was to increase the tensile properties of human hair in order to minimise breakage that is normally prevalent in the braid detangling process.

The hypothesis related to the third objective was that*:*

“Treating human hair with a formulation containing hair strengthening actives will increase the hair’s tensile strength.”

The aim of this part of the study was to study the effects of hydrolysed proteins on tensile strength properties of chemically treated African hair. Increasing the tensile strength of the hair fibres would translate to a reduction in hair damage and reduction in hair breakage during the braid detangling processes. The hypothesis for this objective was accepted. Treating human hair with formulations containing hair strengthening actives was proven to significantly increase the yield strength of the hair.

The safety of the proposed formulation was one of the criteria for an efficient braid detangling formulation. The formulation had to be safe. The proposed formulation is considered to be safe. The individual ingredients used in the proposed formulation adhered to industry regulations. The ingredients were reviewed for safety and regarded as safe for use in cosmetic formulations by the Cosmetic Ingredient Review (CIR) panel.

The optimization of the proposed formulation was also achieved using a d-optimal experimental design. The optimum formulation was found to be one that contained 30% Cyclopentasiloxane , 0% PEG-12 Dimethicone, 10% 18-MEA, 29% water, 10% hair strengthening actives, 12.86% emulsifier combination and 8% other oils. The optimum mixture with the minimum coefficient of friction, predicts a coefficient of 0.61 ± 0.04.

6.2 RECOMMENDATIONS

The study reported in this dissertation was conducted in vitro. It is recommended that the factors such as the braid make-up, hair density and hair texture be taken into account for frictional force studies. It is also recommended that the study of the factors that influence braid detangling be conducted on a large sample pool. The data can be collected using a questionnaire.

6.2.1 Other recommendations

- Study the effects that other factors like, oil build-up , dead hair and dandruff flakes (matting) have on the ease of braid detangling.
- Compare the efficiency of other commercial braid detangling formulations to that of the formulation proposed in this study.