Characterizing the cleaning process of chicken feathers.

A thesis

submitted in fulfilment

of the requirements for the degree

of

Master of Engineering in Materials and Process Engineering

at

The University of Waikato

by

Ganesh Sudalaiyandi

THE UNIVERSITY OF
WAIKATO
Te Whare Wānanga o Waikato

2012

Abstract

Recently, there has been a surge in investigating the potential of using natural fibers for reinforcements in composite materials. One such natural fiber having immense potential for use in polymer composites is chicken feather fiber. Every year, over 4.8 million tonnes of chicken feathers are generated globally. Currently, feathers are hydrolyzed into feather-meal which is used as an animal feed and fertilizer. Chicken feathers are cheap, abundant and easily available. These feathers could be used as reinforcements in polymer composites. But, the feathers obtained from meat processing plants are coated with blood, offal fat, preen oil, debris and poultry processing water. This makes the feathers sticky, odoriferous and unfit for use as reinforcement. Extracting lipids from the feathers by leaching results in fibers which are not greasy and improves the fiber-matrix bonding of composites.

The objective of this study was to characterize the cleaning process of chicken feather fibers. Also, the effect of hydrogen peroxide cleaning on the mechanical properties of feather fiber was tested. The raw feathers were decontaminated with sodium hypochlorite and these samples were used for the cleaning experiments. Cleaning was carried out using 0.15 % and 0.25% of H_2O_2. Stages of cleaning and time were varied. The sample to solvent ratio was 10g/500 ml of solvent. It was found that 10 minutes of leaching for 3 stages was efficient in extracting soluble impurities from the feathers. Equilibrium experiments were conducted and a mass balance based on lipid exchange was designed.An equilibrium graph was plotted. Also, single fiber tensile tests were done on the H_2O_2 treated samples. A two parameter Weibull distribution was plotted to predict the failure strength of the fibers. It was found that H_2O_2 treatment on feathers reduces its Tensile strength (by very less magnitude). It was also observed that fibers were not damaged due to H_2O_2 treatment.

Acknowledgement

First and foremost, I thank my academic supervisor Dr.Johan Verbeek for giving me the opportunity of working under him. I am grateful for all his support, encouragement and patience. I also thank Dr.Mark Lay and Assoc.Prof. Kim Pickering for their academic guidance and being kind to me.

Thank you,Prof. Janis Swan for helping me during my enrolment.

A big thanks to the following technicians:

Alan Smith, Yuanji Zhang, Chris Wang, Lisa Li, Indhar Singh, Wendy Jackson(chemistry dept.) and Helen Turner (SEM facility).

Thanks to Wallace corporation and plant manager Ashley Boon.

I thank Balaji Velram Mohan for his timely advice.

I would like to use this opportunity to thank my dear friends in India for always motivating me and providing moral support.

Finally, I thank my parents and my dear sister for having faith in me and letting me travel my own path.

Contents

1 Introduction **9**

2 Composite Materials **10**

2.1 Introduction to composite materials 10

2.2 Classification of composite materials 10

2.2.1 Laminar Composites 10

2.2.2 Particulate Composites 11

2.2.3 Fibre Reinforced Composites 11

2.3 Types of Fibers 12

2.3.1 Manmade Fibers 12

2.3.2 Natural Fibres 13

3 Chicken Feathers **16**

3.1 Poultry Industry and Rendering 16

3.1.1 Feathers as byproducts 16

3.2 Feather Structure and Types 17

3.2.1 Structure 17

3.2.2 Types of Feathers 17

3.3 Chemical Structure of Chicken Feathers 19

3.4 Application of Chicken Feather Fibers 21

3.4.1 Reinforcement in Composite Materials 21

3.4.2 In Microchips 22

3.4.3 For Filtration of Heavy Metals 22

3.4.4 In Pharmaceuticals and Sanitary Products 23

3.4.5 As Non-Woven Mats 23

3.4.6 Hydrogen Storage 23

3.4.7 Films and Foils 24

3.4.8 Paper and Bullet Proof Vests 24

4 Processing Chicken Feathers **25**

4.1 The need for processing 25

4.2 Steps of Processing 25

4.2.1 Collecting Raw Feathers 26

4.2.2 Washing of Feathers in an Organic Solvent 26

4.2.3 Repeating Washing Step 28

4.2.4 Drying 29

4.2.5 Removing the Fibers from feather shaft 29

5 Leaching and Diffusion **30**
5.1 Steps involved in Leaching 30
5.2 Diffusion 31
5.2.1 Fick's Law 31
5.3 Methods of leaching 32

6 Mechanical properties of fibers **34**
6.1 Introduction to tensile testing 34
6.2 Terms involved 34
6.3 Single fiber tensile testing 36
6.4 Weakest link theory 37
6.5 Weibull distribution 37

7 Experimental **39**
7.1 Materials 39
7.2 Equipments 39
7.2.1 Lamort pulper 39
7.2.2 Boltac mixer 40
7.3 Soxhlet extraction apparatus 40
7.3.1 Instron universal testing machine 41
7.3.2 Image analysis 42
7.3.3 Contherm air forced oven 42
7.3.4 Weighing balance 43
7.4 Experimental design 43
7.4.1 Kinetics experiments 43
7.4.2 Equilibrium experiments 43
7.4.3 Single fiber tensile testing 43
7.5 Methods 44
7.5.1 Decontamination 44
7.5.2 Cleaning 45
7.5.3 Analysis of soluble impurities 46
7.5.4 Equilibrium experiments 46
7.5.5 Separation of fibers from feathers 46
7.5.6 Single fiber tensile testing 47

8 Results and Discussions **48**
8.1 Kinetics experiments 48
8.1.1 0.15 % Hydrogen Peroxide cleaning 48
8.1.2 Impurity evaluation 49
8.1.3 0.25 % Hydrogen peroxide cleaning 49
8.1.4 Impurity evaluation 50
8.2 Equilibrium experiments 52
8.3 Effect of cleaning on morphology of feathers 57
8.3.1 Decontaminated feathers 57
8.3.2 0.15% H_2O_2cleaned feathers 58
8.3.3 0.25 % H_2O_2 cleaned feathers 59
8.3.4 Microscopic morphology 60
8.3.5 Electron microscope images 62
8.3.6 Discussion on surface morphology of treated feathers . . . 64
8.4 Single fiber tensile testing 65
8.4.1 Single fiber morphology 65
8.4.2 Tensile testing 67
8.4.3 Weibull statistics 71

9 Conclusions **77**
9.0.4 Recommendation 77

10 Appendices **87**

A Equilibrium data **88**

B Tensile test data **89**
B.1 10 minutes stage 1 89
B.2 10 minutes stage 3 90
B.3 60 minutes stage 1 91
B.4 60 minutes stage 3 92
B.5 Decontaminated 93

List of Figures

1 Fiber-reinforced plastic roof 10
2 Continuous fibre reinforced composite [10]. 11
3 Discontinuous fiber reinforced composites [10]. 12
4 Feather Structure [35]. 17
5 Types of Feathers [39]. 18
6 Forms of keratin[41]. 19
7 Organization of keratin into fibers[11]. 20
8 steps in leaching [80]. 30
9 Components of solid-liquid extraction in a batch reactor 32
10 Multi-stage countercurrent extraction[84]. 33
11 Example of stress-strain curve 35
12 Diagrammatic representation of single fiber tensile testing[87]. . . 36
13 Lamort pulper 40
14 Boltac mixer 40
15 Representation of soxhlet extraction apparatus 41
16 Instron universal testing machine 42
17 Representation of decontamination process 44
18 Flowchart of cleaning process 45
19 Fibers after separation from feathers 47
20 Leaching of impurities from feather samples treated with 0.15 % H_2O_2. 48
21 Hexane extractable content from feather samples treated with 0.15 % H_2O_2. 49
22 Leaching of impurities from feather samples treated with 0.15 % H_2O_2 50
23 Hexane extractable content from feather samples treated with 0.25 % H_2O_2. 51
24 Quantity of lipids removed from one stage leaching for 10 minutes. 52
25 Quantity of Lipids remaining on the feather samples after leaching. 53
26 Quantity of lipids remaining on the feathers after leaching (calculated). 54
27 Mass balance in leaching of feathers. 55
28 Equilibrium graph 56
29 Decontaminated feathers. 57
30 0.15 % H_2O_2 stage 1 cleaned feathers. 58

31 0.15 % H_2O_2 stage 2 cleaned feathers. 58
32 0.15 % H_2O_2 stage 3 cleaned feathers. 59
33 0.25 % H_2O_2 stage 1 cleaned feathers. 59
34 0.25 % H_2O_2 stage 2 cleaned feathers. 59
35 0.25 % H_2O_2 stage 3 cleaned feathers. 60
36 Surface of decontaminated feathers. 60
37 10 minutes stage 1 60
38 10 minutes stage 2 61
39 10 minutes stage 3. 61
40 60 minutes stage 1 61
41 60 minutes stage 2 62
42 60 minutes stage 3 62
43 Decontaminated feather sample 62
44 10 minutes, stage 1. 63
45 10 minutes 2 stage cleaned feather sample. 63
46 10 minutes 3 stage cleaned feather sample. 63
47 60 minutes 1 stage cleaned feather sample. 63
48 60 minutes 2 stage cleaned feather sample. 64
49 60 minutes 3 stage cleaned feather sample. 64
50 Fiber surface morphology 66
51 A close look at fiber surface morphology 67
52 Tensile stress Vs. Diameter, 10 minutes, stage 1 fibres 68
53 Tensile stress Vs. Diameter, 10 minutes, stage 3 fibres. 68
54 Tensile stress Vs. Diameter, 60 minutes , stage 1 fibres. 69
55 Tensile stress Vs. Diameter, 60 minutes , stage 3 fibres. 70
56 Tensile stress Vs. Diameter, Decontaminated fibres. 70
57 Weibull plot: 10 min. stage 1. 72
58 Weibull plot:10 min. stage 3 72
59 Weibull plot : 60 min. stage 1 73
60 Weibull plot : 60 min.stage 3 73
61 Weibull plot: decontaminated sample. 74
62 Misinterpreted diameter 75

List of Tables

1 Mechanical properties of Man-made fibers [12][13]. 13
2 Mechanical properties of Natural Fibers[19, 20, 21, 22, 23, 24, 28, 25, 26, 27]. 14
3 Mechanical properties of chicken fibers [7, 54, 55, 56]. 22
4 Equilibrium values 56
5 Average values of Tensile stress and Diameters. 71
6 Weibull analysis 74
7 Examples of tensile stress corrections 76

1 Introduction

Composite materials consist of two or more components in such a way that they are insoluble in each other. The matrix confers toughness to the composite and keeps the reinforcement in desired location and orientation[1][2]. The reinforced material is responsible for the strength and stiffness of the composite. Synthetic fibers like kevlar, glass fibers and carbon fibers are very commonly used as fiber reinforcements in composite materials. Natural fibers like coir, hemp, jute and wool have also been used as an effective fiber reinforcement. An alternative natural fiber which can be used as reinforcements in polymer composites are chicken feather fibers[32].

New Zealand has a poultry production of 149 thousand tonnes as of 2011. Poultry meat represents 36 % of the total meat market[3]. The major by-product of the poultry industry is chicken feathers. Currently, feathers are hydrolyzed into feather-meal which is used as an animal feed and fertilizer[4][5]. Much research is being done to explore the alternative applications of chicken feather fibers. Chicken feathers consist about 90% protein, mainly keratin[6]. This confers toughness to the fibers. It has a very low density of 0.8 gm/cm3[7]. It is therefore extremely light and can reduce the weight of the composites manufactured. Feathers have a hierarchal structure; it has a hollow tube like structure called a rachis. The fibers which arise from this rachis are called barbs. The barbs in turn branch out into barbules.

Barbs are the structures in the feathers which can be used as a fiber for reinforcement purposes. Feathers obtained from the meat processing plants are coated with blood, offal fat, preen oil, debris and poultry processing water. This makes the feathers sticky, odoriferous and unfit for use as reinforcement fiber. Therefore, cleaning the feathers is required in order to use the fibers for reinforcement purposes.

The objective of this study is to characterize the cleaning process of chicken feather fibers using hydrogen peroxide as a solvent and then observe and analyze the effect of H_2O_2 cleaning on the morphological and mechanical properties of the feather fibers.

2 Composite Materials

2.1 Introduction to composite materials

Composite materials consist of two or more materials (as components) in such a way that they are insoluble in each other. This makes the composite materials stronger than they are apart [1][2]. One of the earliest examples of composites dates back to 1500 B.C when the Egyptians used straw to reinforce mudbricks. Ever since, composite materials have found a place in this world and are typically used in household appliances, automobiles, construction industry, electrical appliances, marine industry, aircraft industry and many more [8][9].

A very common example of a composite material used widely is a fiber-reinforced plastic roof. It's structure is depicted in the following diagram:

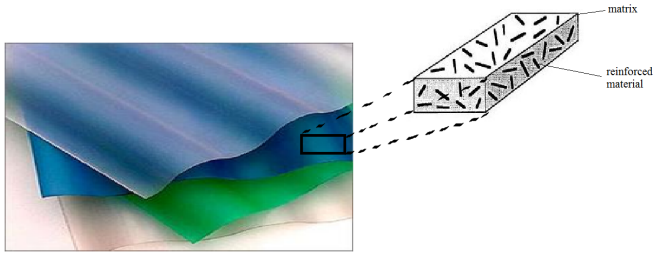


Figure 1: Fiber-reinforced plastic roof

It can be noticed from the figure that the plastic roof is composed of a matrix and a reinforced material. The matrix confers toughness to the composite. It keeps the reinforcement in desired location and orientation. Whereas, the reinforced material is responsible for the strength and stiffness of the composite [11][2].

2.2 Classification of composite materials

There are three main types of composites depending on the reinforcements used [11].

2.2.1 Laminar Composites

Laminar composites comprise of layers of materials bonded together. There can be two or more layers of materials arranged in a determined order. Examples

include plywood and modern skis.

2.2.2 Particulate Composites

Particulate composites comprise of particles distributed or embedded in a matrix body. The strength of the composite usually depends on the diameter of the particles, the inter-particle spacing, volume fraction and properties of the matrix. Concrete is a very well known example of a particulate composite. The stone is the particle and cement acts as a matrix.

2.2.3 Fibre Reinforced Composites

In the type of composites, fibers are used as the reinforcement material on a plastic resin. A fiber has a high length to diameter ratio. This is known as an 'aspect ratio'. There are two major types of fiber reinforcements.

- Continuous fibre reinforcements

These fibers have very high aspect ratios and generally have a preferred orientation. Any further increase in the length of the fiber will not change the properties of the composite. Continuous fiber composites are used where higher strength and stiffness are required but at a higher cost. A diagrammatic representation of continuous fiber reinforcements is shown below in Fig.2.

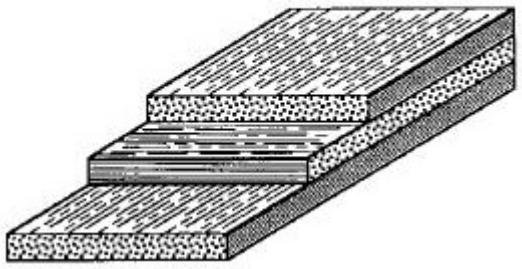


Figure 2: Continuous fibre reinforced composite [10].

- Discontinuous fibre reinforcements

The fibers have low aspect ratios and normally have a random orientation. The applications involving multi-directional applied stresses commonly use discon-

tinuous fibers. Also, it is cheaper to manufacture such composites. A diagrammatic representation of discontinuous fiber reinforcements is shown below in Fig. 3.

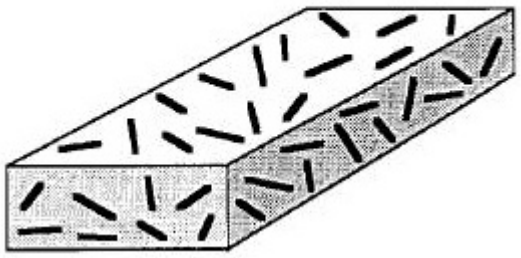


Figure 3: Discontinuous fiber reinforced composites [10].

Fiber-matrix bonding is an important factor which affects the strength of a composite material in both continuous and discontinuous fiber reinforced composites. Good adhesion of the fibers to the matrix increases the strength of a composite material. However, the final properties of a composite material manufactured depends on factors such as mechanical properties of the fiber, type and orientation of fibers, volume fraction of fibers and processing techniques used.

2.3 Types of Fibers

2.3.1 Manmade Fibers

Man-made fibers are synthesized from polymers. The raw materials used to manufacture the polymers are derived from petrochemicals. Large number of consumer and industrial products have man-made fibers incorporated in them. Typical examples include automobile bodies, sports equipment, smart phones, thermal insulation, in cables and many more. Some of the most widely used synthetic fibers are kevlar fibers, glass fibers, carbon fibers and aluminum fibers. The advantages of using synthetic fibers are high tensile strength, stiffness, thermal stability, large scale manufacturing, to name a few. Mechanical properties of some manmade fibers are listed in Table 1:

Table 1: Mechanical properties of Man-made fibers [12][13].

Fiber	Density(g/cm^3)	Diamet.(μm)	E-failure(%)	Ten.strength(Mpa)	E-mod.(GPa)
E-glass	2.5	9-15	2.5	1200-1500	70
carbon	1.4	5-9	1.4-1.8	4000	230-240
aluminium	2.7	20-200	0	110	69
kevlar 49	1.44	-	2.4	3800	131

2.3.2 Natural Fibres

Natural fibers are obtained from plant and animal sources. Plant fibers include stem, leaf, seed, fruit, wood, cereal straw and other soft grass fibers. Animal fibers include hair, wool and silk. Plant fibers are composed of cellulose while animal fibers consist of proteins. A quick glance at the types of natural fibers will reveal its versatility [14][15][16]. The mechanical properties of some popular natural fibers are listed in Table. 2.

Fibres from Fruit

Cotton

Cotton is the most used textile fiber in the world. It is almost pure cellulose. It is cultivated as an annual shrub. The cotton fiber grows in a boll around the seeds of the cotton plant. It is primarily used in textiles, mattresses, fishing nets, to name a few [16][17].

Coir

The fiber from the outer layer of coconuts is called as coir. The fibrous layer of the fruit is separated from hard shell. The husk is soaked in water for about 10 months (retting) and then beaten to break away the fibers. The coir fibers are strong, light in weight and can withstand heat and salt water. The applications of coir include brushes, mattresses and geotextiles [16][17].

Fibres from Stem

Plants like flax, hemp, jute, kenaf, ramie bamboo fall under this classification of fibers. These plants are cut from the ground and soaked in water, then stripped from the core and dried. These fibers are utilized in fabrics, bags, canvas, cordage, carpeting and non-wovens [14][16][17][18].

Fibres from Leaves

Sisal and abaca are the most important leaf fibers. Sisal is a drought tolerant plant which has rosettes of long, pointy, fleshy leaves. It is used in carpets, composite materials and paper pulp. In abaca, the leaves are first harvested and then boiled to prepare the fibers. Applications of abaca fibers include a range of specialty papers, sausage casings, tea bags, coffee filters and bank notes [14][16][17][18].

Table 2: Mechanical properties of Natural Fibers[19, 20, 21, 22, 23, 24, 28, 25, 26, 27].

Fiber	Den.(g/cm^3)	Dia.met(μm)	E- failure(%)	Ten.strength(MPa)	E-mod(GPa)
Bagasse	-	490	-	70	-
coir	1.2	-	30	175	4-6
Cotton	1.5-1.6	20	7-8	287-597	5-13
Curaua	1.38	66	3.9	913	30
Flax	1.5	50-100	2.7-3.2	342-1035	50-70
Hemp	1.10	120	1.6	389-900	35
Henequen	-	180	3.7-5.9	430-570	10-16
Jute	1.3	260	1.5-1.8	393-773	26
Kenaf	1.31	106	1.8	427-519	23-27
Pineapple	1.32	-	2.4	608-700	25-29
Ramie	1.50	34	3.6-3.8	400-938	24-32
Sisal	1.5	50-80	2-2.5	337-413	8-10
Bamboo	0.88-1.10	100-200	-	391-713	18-55
Silk	1.34	3.57-12.9	18-40	260	9.9
Spider silk	1.30	3.57	17-18	875-972	11-13
Wool	1.31	16-40	25-35	120-174	2.3-3.4

Animal Fibres

The most widely used animal fibers are wool and silk. Wool is the fiber from the fleece of sheep and similar hairy animals. Examples include alpacas, lalmas, vicunas, yaks, camels cashmere goats and angora rabbits. It is primarily used for coarser types of bedding, upholstery and carpets. Silk is produced from the cocoons of silkworm. The silkworm secrets an unbroken fiber cocoon which is boiled and unbound to form the fiber which is then spun. Silk has applications in textile industries.

These natural fibers have an enormous potential to replace conventional man-made fibers. They have low cost, low resource consumption for their production, good mechanical properties, non-abrasive, renewable, have low density, are biodegradable and can be recycled [14, 16, 17, 18].

3 Chicken Feathers

3.1 Poultry Industry and Rendering

The global poultry industry includes chicken, turkey, duck, guinea fowl , goose, quail, pheasants and squabs, but chicken meat has dominated the global poultry industry since many years [29]. In 2011, the world chicken meat production amounted to 89.2 million tonnes [30]. New Zealand has a poultry production of 149 thousand tonnes. Poultry meat is the most popular meat in New Zealand representing 36 % of the meat market [3].

The poultry farms rear meat chickens called broilers for 42-45 days. They are then slaughtered and processed. The first step of slaughtering is stunning and killing, followed by scalding and then defeathering [31]. Feathers are removed by mechanical pluckers fitted with rubber fingers. Defeathering is completed by operators called pinners, who manually finish plucking. The feathers are pumped over a separation screen into a container with a mixture of dilute blood, grease, cleaning water and feathers [5]. After defeathering, removal of heads and legs follows. Heads, beaks and feet are often mixed with the feathers. The next step is evisceration. Here, the viscera (internal organs) are removed. It can be done either with knives (manually) or by using complex, fully automated mechanical devices. The carcasses are inspected during the evisceration process.

After the carcass has been washed, they are chilled to a temperature below 4°C. Later, whole or individual parts of the birds may be packaged raw for direct sale. They may also be further be processed into deboned and ground meat [32].

3.1.1 Feathers as byproducts

Such an intensive meat production has also led to generation of over 4.8 million tonnes of chicken feathers world wide every year [42, 7, 33, 34]. Chicken feathers are often seen as a waste and the most widely used methods for disposal of chicken feathers are land filling and burning [7, 33, 34]. Feathers, blood and offal are rendered into a high protein product called feather meal. Amino acid supplements are added to improve feed quality. This product is 60% digestable [4, 5]. Researchers all over the world are exploring opportunities to increase revenue by developing alternative applications for chicken feathers. But, before implementing these applications, it is necessary to know the physical and chemical structure of the chicken feathers.

3.2 Feather Structure and Types

3.2.1 Structure

Chicken feathers have a central, hollow tube like structure called a rachis. From this rachis arises feather fibers called barbs. Technically, feathers have a hierarchical and branched structure (shown in Fig.4) and they can be divided into three parts:

- rachis
- barbs
- barbules

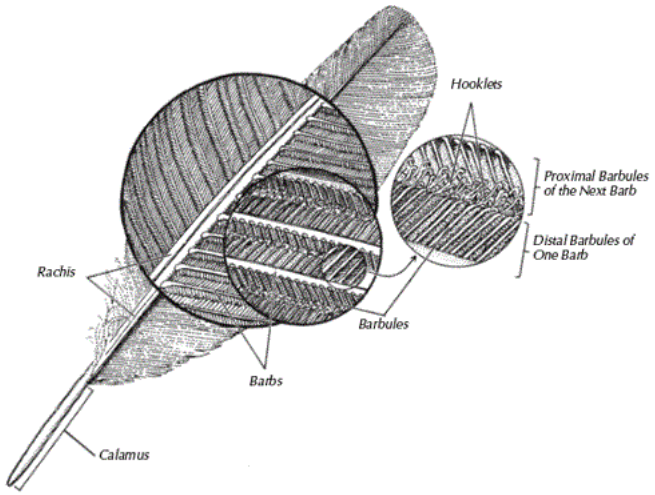


Figure 4: Feather Structure [35].

The barbs are upto 35 mm long and have a diameter of 40-400μm. The barbules have lengths of less than 1 mm and diameters of 10-30μm. Barbules have hooks known as barbicels, which connect with barbules on adjacent barbs. The rachis length is typically between 40-120 mm and diameter can exceed 3mm [7, 36].

3.2.2 Types of Feathers

The primary function of feathers is for flight and protection of the skin of the birds. In the domesticated poultry, feathers grow in distinct tracts. They grow

all over the body of a chicken and they appear to have a uniform feather cover [37]. Feathers constitute 3-6 % of the body weight of the chickens. There are 5 main types of feathers based on morphology [38]. It is shown in Fig.5.

- contour feathers
- downy feathers
- semiplume
- filoplume
- bristle

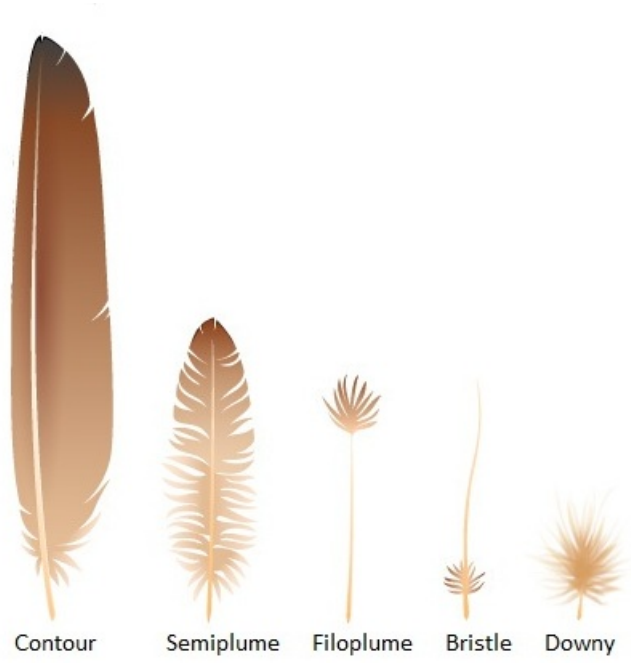


Figure 5: Types of Feathers [39].

Contour feathers are primarily responsible for flight and provide defense against physical objects, sunlight, wind and rain. The downy feathers are smaller than the contour feathers and lack barbules and barbicels. They are soft and fluffy, located beneath the contour feathers. They provide most of the insulation to the birds. Semi-plumes have characteristics of both contour and downy feathers. They have long rachis and barbs similar to downy feathers.

Filo-plumes are smaller than semi-plumes, with only a few barbs at the tip of a fine shaft. They serve a sensory function in a chicken, registering vibrations and changes in pressure. Bristles are stiff and have very short barbs near the tip. Bristles are protective in function and are found on a chickens head, at the base of the beak, around the eyes and covering the nostrils[40].

3.3 Chemical Structure of Chicken Feathers

Nearly 90 % of the barbs and the rachis of the feathers are made up of a protein called keratin. Keratin is insoluble and highly durable protein found in hair, hoofs and horns of animals[6]. Based on structure, there are two different forms of keratin. It is shown below in Fig.6.

- alpha helical structure of keratin
- beta pleated sheet structure of keratin

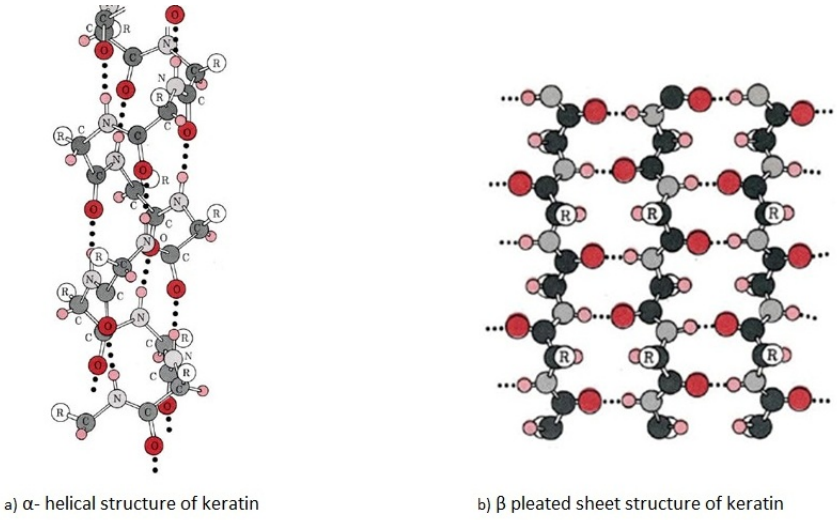


Figure 6: Forms of keratin[41].

The barbs and the barbules of the feathers have the alpha-helical structure of the keratin. Feather keratin has a molecular weight of about 10,500 g/mol and cystine content of 7%. Cystine is responsible for the sulfur-sulfur bonding in keratin[42]. The fibrous keratin molecules supercoil to form very stable, left handed super helices. On the other hand, the central portion in the feather,

the rachis, is rich in hydrophobic residues and predominantly has beta-pleated sheet structure of keratin[43].

The polypeptide chains of keratin arrange into protofilaments. Many of these protofilaments join together to form microfibrils. These fibrils themselves become attached to form even thicker and denser fibrils. The cell contents then dehydrate and become replaced with other keratinous fibrils. In this way the keratins are organized into fibers[44]. This diagrammatic representation of this process is shown in Fig.7.

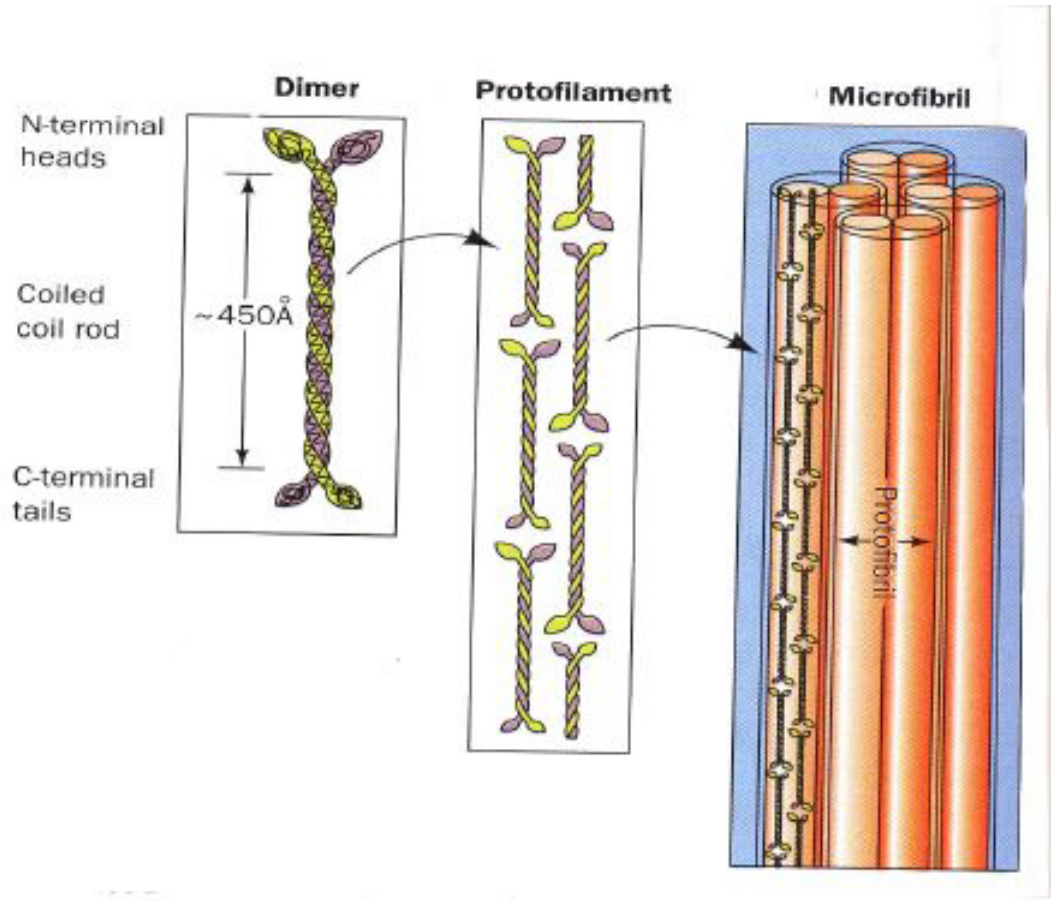


Figure 7: Organization of keratin into fibers[11].

The chicken feather fibers are rigid in structure due to the hydrogen bonding of the helix-proteins. The di-sulfide bonds of cystine stabilizes the cylindrical units into very strong cables[45]. The amino acid sequence of keratin in chicken

feathers is similar to the amino acid sequence of keratin in other birds. It also has a great deal in common with other reptilian keratins from claws. The sequence is largely composed of cystine, glycine, proline and serine. Where as methionine, histidine and lysine are almost absent[46].

3.4 Application of Chicken Feather Fibers

Chicken feather fibers are cheap, biodegradable and a great source of small diameter, high surface area, tough and durable fiber making them attractive for use in different industries[47]. There have been numerous studies conducted on the uses and applications of chicken feather fibers, some of which are discussed below.

3.4.1 Reinforcement in Composite Materials

The unique shape of the feather fibers, a center fiber with many branching fibers, makes feathers ideal for random orientation processes such as injection molding, dry formation or wet laying[48].

The chicken feather fiber diameter is approximately 5-50μm and its length ranges from 1 mm to 35 mm [21, 37, 40, 41]. The length of the fibers is an important consideration as it would affect the stress transferability between the matrix and the fiber[50]. The bonding between the polymer matrix and the fiber is an important factor affecting the quality of composite manufactured.

The presence of rachis in the chicken feathers makes it more granular, bulkier, light weight material. Removal of the rachis results in smoother and denser products. The rachis is preferable to be used as fillers. Hence, for most of the fiber-reinforcement work, the fiber is cut from the rachis of the feathers[51, 52].

Chicken feather fibers have good mechanical properties and are non-abrasive, making them ideal for use as reinforcements in polymer matrices. Examples include interior panelling, ready to assemble furniture in automobiles, trucks, homes, offices and factories. In composites with thermoset polyesters, feathers were reported to increase strength by 20% and decrease weight by 50%[53]. Some properties of chicken fibers are summarized in Table 3.

Table 3: Mechanical properties of chicken fibers [7, 54, 55, 56].

Density(g/cm^3)	0.8
Tensile strength (MPa)	190-203
Youngs modulus	3.6-4.5
Melting point (^{0} C)	240

In terms of fiber reinforcement, the use of down feather fibers is found to give better results than flight feather fibers. This is because, the flight feather fibers have hollow structures while the down feather fibers have solid structures[50].

The down industry separates whole feathers into sizes to extract the finest feathers from other feathers. The separation of the down feathers from a mixture is based upon the principle that smaller, light weight features have greater lift in an upflow of air than larger feathers[51].

3.4.2 In Microchips

A proposed application of chicken feathers is in computer chips[57]. Hong,C.K and Wool,R.P [55] have investigated the potential of including chicken feathers in circuit boards to replace silicon, creating bio-based microchips. These biobased microchips have some advantages over the conventional microchips.

Chicken feathers have a very low density. They are light and tough. Thus, they provide strength and decrease the weight. The dielectric constant of air is 1.0 and that of silicon di oxide 3.8-4.2, where as keratin fibers have a dielectric constant of 1.6. Which means that electrons can move on feather based circuit boards at twice the speed as traditional circuit boards.Prototypes of this chicken feather microchips are being trailed[57] .

3.4.3 For Filtration of Heavy Metals

Chicken feathers were sucessfully used as a biosorbent for heavy metal removal from aqueous solutions. Heavy metals like lead, arsenic, compounds like phenols and few hazardous dyes were reported to be filtered from water using chicken feathers. The need to develop effective , low-cost and environmentally friendly methods for water treatment has lead to the selection of chicken feathers for biosorption.

High tensile strength, water insolubility, structural toughness , stability over a wide range of pH are the reasons for chicken feathers being preferred. Chicken

feathers are also used in packed columns for filtration applications for a number of adsorption/desorption cycles[59, 60].

3.4.4 In Pharmaceuticals and Sanitary Products

Keratin of poultry feathers and its derivative nano-composites have the potential to be used in biomedical products intended for wound repairing and bone tissue regeneration. It can be in skin care products, as it makes the skin, hair and nails healthy. This is because of it's high sulfur content which contributes to the growth of cartilages, bone tissues, tendons, hairs, nails and skin[61].

There is a demand for textile materials having specific properties like the ability to absorb and retain humidity. Biomodified cellulose and keratin obtained from poultry feathers were used to make composite sanitary products. They have advantages of better sorption properties, higher hygroscopicity and smaller wetting angle when compared to cellulose fibers[61].

3.4.5 As Non-Woven Mats

Non-woven mats are sheet like structures formed by mechanically, thermally or chemically intertwining fibers. They are not made by weaving or knitting. Feather fibers have been utilized to make non-woven mats. These mats are used to prevent soil erosion during the re-vegetation process of several land restoration projects. The mat can survive in the soil for 2 years and degrades almost completely[62]. Ye and Broughton[63] created a non-woven mat which was reported to exhibit better insulating properties than polyester fibers. Chicken feather fibers are reported to ideal erosion control agents.

3.4.6 Hydrogen Storage

Hydrogen is a leading alternative fuel for vehicles. Storing sufficient quantities of hydrogen under normal pressure is very difficult. Carbon nano tubes are the ideal storage units of hydrogen under normal pressure. But, a carbon nano tube storage tank is extremely costly. Researchers have found that carbonized feathers can store as much hydrogen as carbon nano tubes. Moreover, storage tanks made of carbonized feathers would be very cheap to manufacture. Chicken feathers could be a promising solution to tackle the hydrogen storage problem[64].

3.4.7 Films and Foils

Treatment and softening of keratin in chicken feathers leads to the formation of translucent biodegradable films and foils. It could possibly be made use of as a plastic wrap/food wrap in future. The film thus produced can also be used as a drug encapsulation material. Further research in the field is being conducted[65].

3.4.8 Paper and Bullet Proof Vests

Feather fibers are useful in making air-filters and decorative papers. This could help in decreasing the amount of wood pulp used for paper production. Air filters made from feather fibers have smaller and more pores. It could provide finer filtration[66].

A British defense R&D project has experimented making bullet proof vests from a fabric created from woven feather quills. The product was reported to be lighter and more comfortable than kevlar vests and seemed to offer excellent protective qualities. It is also less expensive[66].

4 Processing Chicken Feathers

4.1 The need for processing

Chicken feathers collected from meat processing plants should be processed for the following reasons:

In meat processing plant, feathers are plucked from chicken. The meat is packed and the feathers often lie as heaps mixed with offal, dilute blood, grease and water. Unprocessed raw feathers appear straw-like, with a greasy texture and the barbs are stuck to the rachis.The feathers obtained/collected are discolored and have obnoxious odor[5].

The freshly collected feathers could possibly be harboring a variety of disease causing micro-organisms. The common genera of microbes which are found on the raw chicken feather fibers are campylobacter, enterobacter, salmonella and Escherichia. These pathogens are known to cause gastroenteritis[67, 68, 69].

In section 2.2.3, discontinuous fiber reinforcements were discussed. The chicken feather fibers (short fibers) are mostly used as discontinuous fiber reinforcements. By processing and cleaning chicken feather fibers it is made sure that the fiber surface is freed from lipids and fatty acid coatings. Cleaning of lipids and fatty acids from the feather surface enhances the bonding between the matrix and fiber. It improves the stress transferability between the matrix and fiber and thus increasing the quality of the composites manufactured. Processing of freshly collected feathers prevents it's decay. The processed feathers can be stored safely at room temperature.

4.2 Steps of Processing

Processing chicken feathers typically requires several steps. A United States patent by Gassner et al describes the main steps as follows [52]:

- collecting raw feathers
- washing the feather in an organic solvent
- repeating washing step
- drying the feathers
- removing the fibers from feather shaft

4.2.1 Collecting Raw Feathers

The raw feathers are collected from meat-processing plants in air tight containers and then transported to the feather processing units. The feathers obtained from the meat processing plants have a combination of all the types of poultry feathers. It is a more practical way to use all these feathers for composite material production. The collected feathers should be processed as soon as possible to prevent its decay[42, 7].

4.2.2 Washing of Feathers in an Organic Solvent

The feathers are washed in organic solvents to remove impurities like preen oil, offal fat from feather surfaces and also to whiten the fibers. The organic solvents used to wash feathers should have the following features:

- The solvent should be able to extract the target compounds in a short time.
- The solvent should be compatible with the sample and should not react with target compounds.
- The solvent should be chemically and thermally stable during operational conditions.
- Low viscosity is necessary to increase the diffusion co-efficient and to keep the extraction rate higher.
- Low flammability.
- Low toxicity.
- environmentally friendly.

Ethanol is an organic solvent used for cleaning chicken feathers, patented by Gassner et al. After the cleaning, ethanol is distilled and re-used. When organic solvents are used for cleaning, their discharge could cause potential problems for the environment. Inorganic solvents like hydrogen peroxide, sodium hypochlorite and detergents like sodium dodecyl sulfate (SDS) can be used for cleaning[70, 71]. A brief look into a few inorganic solvents would show their significance:

Sodium Hypochlorite

Liquid sodium hypochlorite is the most widely used bleach. It performs three important functions[72].

- oxidizes and aids in removal of dirt
- acts as a disinfectant
- whitens the fibers

Sodium hypochlorite (1-5% w/v at pH 10 to 12) can be used to make the raw unprocessed feather bacteriostatic. It can be sucessfully used as a decontaminating agent[73]. Decontamination of chicken feathers using sodium hypochlorite needs to be done at a pH above 10, since it's active component can exist in three different states.

- at a pH greater than 10, the hypochlorite is present as sodium hypochlorite.
- at pH 5 and 8.5 the solution consists predominantly of hypochlorous acid and as the pH falls below 5, the liberation of chlorine takes place.
- when pH falls below 3, all the hypochlorous acid is converted into chlorine.

$$HOCl \rightleftharpoons H^+ + OCl^-$$

$$HOCl + H^+ + Cl^- \rightleftharpoons Cl_2 + H_2O$$

Hydrogen Peroxide

The objective of using hydrogen peroxide is to bleach the fibers.

Hydrogen peroxide (3 % concentration) is a well-known bleaching agent.

The active oxidizing agent is the perhydroxyl ion species [74].

$$H_2O_2 \rightleftharpoons H + HO^{-2}$$

Hydrogen peroxide is a favorable bleaching agent when using protein fibers, since it does not react with proteins. This aids in retaining the mechanical

properties of the feather fiber. Treating keratinous fibers with hydrogen peroxide leads to breakdown of cystine linkages. A considerable number of disulfide linkages are first hydrolyzed and later oxidized to varying degrees [74, 75].

$$R.SS.R \rightarrow R.SH + R.SOH \rightarrow R.SO_2H$$

Feathers contain melanin pigments (These pigments provide colour to the feathers). It is suggested by few researchers that during bleaching of a fiber by hydrogen peroxide, it interacts preferably with melanin discs and less with keratins of fibers [76]. This may be the reason for whitening of the feathers after bleaching and yet retaining good mechanical properties.

Hydrogen peroxide also performs the function of lipid removal. It is used clinically to remove ear wax. A brief look about the mechanism of H_2O_2 in removal of ear wax could be helpful to understand its lipid removal action. The majority of the epidermal cells in the ear are keratinocytes. The ear wax contains keratinocytes and secretions like long chain fatty acids, alcohols, squalene and cholestrol. When applied, H_2O_2breaks up the ear wax through the release of gas/bubbles (mild foaming) due to its reaction with enzyme catalase. The wax is removed due to this mechanical action [77, 78]. A similar mechanism may be involved in removal of lipids from the surface of feathers, since keratinocytes and lipids are involved in both the cases. It is environmentally friendly to use hydrogen peroxide because it breaks down to water and oxygen after the treatment and is safe for disposal [79].

4.2.3 Repeating Washing Step

In most cases, washing needs to be more than once. The number of times washing is repeated depends on the desired level of cleaning. It could be a one time wash for a long duration or divided into several short spells. The former may require more time and less solvent, whereas the latter may require less time and more solvent.

The amount of solvent used depends on the solvent to the feed (here, chicken feathers) ratio. High solvent to feed ratio means higher solvent consumption. It is related to the economic aspects of the concerned industry [80].

4.2.4 Drying

Chicken feathers recovered after cleaning are dried to constant mass using hot air oven. Wet feathers are fluffed before drying to disperse the particles. Greater surface area facilitates efficient drying. After drying, the feathers can be compared to the unprocessed raw feathers to observe the significant change in feather color and texture.

4.2.5 Removing the Fibers from feather shaft

In section 2.4.1, the reason for stripping of the fibers from the rachis of the feathers for commercial production of fibers was mentioned. This process is called as comminution. The principle involved here is application of mechanical stress on the feathers to reduce the particle size. The equipment used for this process can be refiners, pulpers or disc mills. A patent on comminution states that refiners or disc mills grinded, sheared, shredded, pulverized, rubbed and fluffed the feather into short fibers. The fibers recovered are dried to constant mass and then labeled and packed. These fibers are ready to be used as reinforcements for various polymer matrices [19].

5 Leaching and Diffusion

5.1 Steps involved in Leaching

Leaching is the removal of a soluble fraction of a solid material by a liquid solvent. The solute diffuses from inside the solid into the surrounding solvent. Leaching is also termed solid-liquid extraction. Either the extracted solute or the insoluble solid portion may be the valuable product[81, 82].

The general steps involved in any solid-liquid extraction are:

1. Solvent is transferred from bulk solution to the surface of the solid.
2. Solvent penetrates into the solid.
3. Solute dissolves from the solid into the solvent.
4. Solute diffuses to surface of the solid.
5. Solute is transferred to bulk solution.

These steps are represented as a diagram in the following Fig.8:

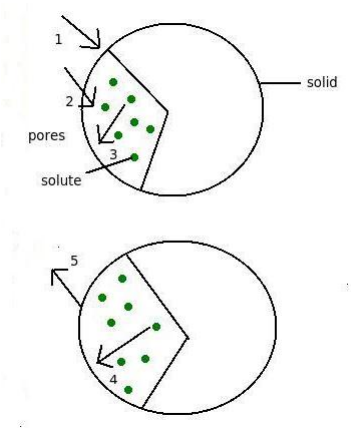


Figure 8: steps in leaching [80].

The first step of leaching, that is, the transfer of the solvent from bulk solution to the surface of the solid takes place rapidly. The rate controlling process is the diffusion of the solvent into the solid. This varies and it depends on the structure of the solid feed.

5.2 Diffusion

Chicken feathers are heterogenous structures. The feather fiber has a semi-crystalline nature and the particle size of the feathers used for leaaching are highly non-uniform. This makes the penetration of the solvent into chicken feathers tough and leads to irregularities in diffusion. The diffusion process is very slow when biological systems are involved, since they have complex membranes and cell structures[83].

5.2.1 Fick's Law

The phenomenon of diffusion is explained by Fick's law[80, 83].

The rate of mass transfer of a solute B which is dissolved to a solution of volume V is given by:

$$N_B = K_L A(C_{BS} - C_B) \tag{1}$$

where,

N_B= Kgmol of B dissolving to the solution

A = Surface area of particles (m^2)

K_L= Mass transfer co-efficient (m/sec)

C_{BS}= Saturation solubility of the solute B in the solution in Kgmol/m^3

C_B = Concentration of B in the solution at time t seconds in Kgmol/m^3

The rate of accumilation of B in the solution is equal to the dissolving flux:

$$\frac{VdC_B}{dt} = N_B = K_L A(C_{BS} - C_B) \tag{2}$$

Integrating from t=0 and C_B= C_{B0} to t=t and C_B=C_B

$$\int_{C_{B0}}^{C_B} \frac{dC_B}{C_{BS} - C_B} = \frac{K_L A}{V} \int_{t=0}^{t} dt \tag{3}$$

$$\frac{C_{BS} - C_B}{C_{BS} - C_{B0}} = exp(-\frac{K_L A}{V} t) \tag{4}$$

The solution approaches saturated condition exponentially.

Effective diffusivities in solids depends on molecular forces, solubility, cell structure, volume fraction, concentration and temperature.

5.3 Methods of leaching

The method of contacting solids with solvent is either by percolation of solvent through a bed of solids (or) by immersion of the solid in the solvent followed by agitation of the mixture. When percolation is used, either a stage wise or a differential contacting device is appropriate. When immersion is used, countercurrent multistage operation is commonly followed. Thus, the equipments used to conduct leaching can be under batch, semi-continuous or continuous operating conditions.

When the solids to be leached are in the form of fine particles (smaller than 0.1 mm in diameter), then batch leaching is followed. The process is conducted in an agitated vessel. The leaching process in a one stage batch reactor is shown in the following diagram:

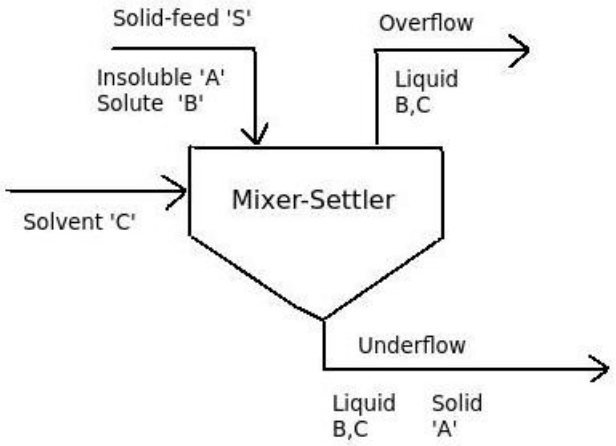


Figure 9: Components of solid-liquid extraction in a batch reactor

The components of a leaching system are highlighted in the above figure. The effluents are a key component and play an important part in leaching. They are:

- Overflow - it is the solid-free liquid.
- Underflow - it is the wet solids (or) slurry stream.

When the solids to be leached are too coarse, percolation techniques are used. Here, the solids which need to be leached are dumped into a vessel and then a

solvent is added for percolation through the bed of solids. In order to achieve a high concentration of solute in the solvent, a series of vessels is arranged in a multi-batch, countercurrent leaching technique. An example is the Shank's extraction battery.

When leaching is carried out on a large scale, it is preferable to use an extraction device that operates with continuous flow of both solids and liquid. Examples of such continuous extractors are: Bollman extractor, Rotocel extractor and continuous perforated belt extractors.

When, there is a need to reduce the concentration of the solute in the liquid portion of the underflow, leaching is carried out in countercurrent type of extraction. The Fig.10 depicts the processes involved in a multi-stage countercurrent extraction.

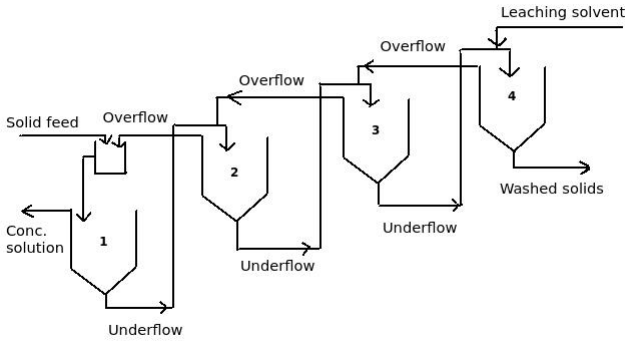


Figure 10: Multi-stage countercurrent extraction[84].

When the leaching rate is slow, several countercurrent stages may be employed. It is therefore called as multi-stage countercurrent extraction. In this system, the solid feed and the solvent are fed in opposite directions. The separated solids and liquid move countercurrently to adjacent stages. The solvent phase (or) extract becomes increasingly concentrated as it contacts solid feed, in a stage wise manner. Also, the raffinate becomes less concentrated in soluble material as it moves toward the fresh solvent phase. Using this principle, it is theoritically possible to reduce the solute contact of the raffinate by increasing the number of the stages/contact.

6 Mechanical properties of fibers

6.1 Introduction to tensile testing

Any material which is to be used in structural applications requires it's mechanical properties to be tested. By doing so, the properties of materials used can be determined and it enables in the selection of appropriate materials. The properties of materials reported in various handbooks are the results of such tests. One of such tests is the tensile test.

It is the most fundamental type of mechanical test that can be performed on a material . The tensile test measures the resistance of a material to a static (or) slowly applied force. The forces includes a pulling (or) stretching force (Force = F, called Load). The amount of force required to break a material and the amount it extends before breaking are measured through this test and it constitutes the mechanical properties of the test object[85].

6.2 Terms involved

Breakage (or) failure of a sample can occur either due to excessive stress (or) excessive deformation. For most materials the initial resistance to force and the point of permanent deformation are obtained from the plots of force against elongation (or) stress-strain curve[2].

$$stress(\sigma) = \frac{F}{A_0} \tag{5}$$

$$strain(\varepsilon) = \frac{l - l_0}{l_0} \tag{6}$$

where,
F = force applied
A_0 = original cross-sectional area of the specimen.
l_0= original distance between the gauge marks.
l = distance between the gaugemarks after force is applied.

An example of a typical stress-strain curve is shown below in Fig.11:

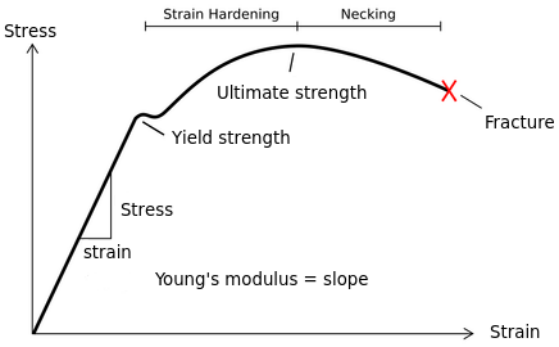


Figure 11: Example of stress-strain curve

Analysis of stress-strain curves can predict the behavior of a test material under a certain force. There are many testing machines to conduct this analysis. The most common are the universal testing machines, which test materials in tension, compression (or) bending[86]. A stress-strain diagram is created which can be used to calculate yield strength, Young's modulus, tensile strength and total elongation.

- The stress applied to the material at which plastic deformation becomes noticeable is called as the yield strength of that material.
- The Young's modulus or the modulus of elasticity (E) is the slope of the stress-strain curve in the elastic region. This relationship is the hook's law[2].

$$E = \frac{\sigma}{\varepsilon} \tag{7}$$

- Tensile strength is the maximum load which a specimen can bear during the test. It may or may not equate to the strength at failure.
- Elongation (%) The extent of stretching which a material can withstand without breaking is measured here.

$$\%elongation = \frac{l_f - l_0}{l_0} \times 100 \tag{8}$$

where,

l_0= initial distance between gauge marks.

l_f= distance between gauge marks after the sample breaks.

6.3 Single fiber tensile testing

Single fiber tensile test is most widely applied method for measuring the tensile properties of individual fibers. In this method, single fibers are mounted on special slotted tabs and loaded on tensile testing machine where stress is applied on the samples[88]. Tensile strength, young's modulus and failure strain of single fibers are determined through this method. A diagrammatic representation is shown in Fig.12.

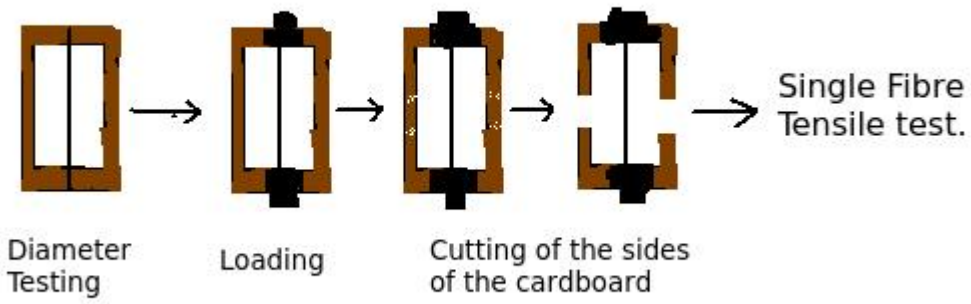


Figure 12: Diagrammatic representation of single fiber tensile testing[87].

The cross-sectional area (A) of the specimen is measured before the test. After the specimen is mounted on the test machine, the center section of the tab is cut away to allow for fiber elongation. ASTM D3379-75 is the standard test method for single fiber tensile testing[88]. The strength of the fiber is measured by :

$$\sigma_f = \frac{P_{max}}{A} \tag{9}$$

where,

P_{max}= maximum load

A = cross-sectional area

By measuring the slope of the linear portion of the stress-strain curve, the Young's modulus is calculated.

6.4 Weakest link theory

In the earlier sections, it was mentioned that the cross-section (or) diameter (d) of a single fiber is measured before tensile testing. The natural fibers do not have constant diameter throughout it's length. This may lead to discrepancies predicting the tensile strength of the material. It was proposed by Griffith that the fracture of a specimen begins at it's flaw center and the propagation of this crack leads to the fracture of that material. The weakest point in a fiber could be its flaw center. It could also have a very small diameter. If this weakest point reaches its breaking limit, then the entire fiber breaks. Also, there is a length correlation. The longer the fiber the more likely it is to have a severe flaw and will therefore will be weaker. This concept is known as weakest link theory[89].

6.5 Weibull distribution

If the strength of fibers need to be modelled accurately, then the distribution of fiber strengths needs to be included in the model too. Weibull distribution is used for this purpose. It describes the failure rate and wearing out of materials. It is named after a Swedish physicist, W. Weibull. Based on the weakest-link theory, the Weibull distribution is widely used to describe the tensile strength of synthetic fiber materials.

It is expressed by the formula:

$$P_{f(L)} = 1 - exp[-n(\frac{x - x_\mu}{x_0})^w] \tag{10}$$

where x_0 is the characteristic strength of a unit length for which the probability of failure is $0.632(1 - exp(-1))$, also known as the scale parameter. w is the shape parameter or Weibull modulus and $x_\mu$is the lowest value for strength and is often set to zero for simplification.

$P_{f(L)}$is the probability of failure of a fiber of a length L.

The above mentioned formula can be further simplified as follows:

$$P_{f(L)} = 1 - exp[-L(\frac{\sigma}{\sigma_0})^w] \tag{11}$$

This is known as the Weibull two parameter cumulative distribution function. This expression can be further rearranged to produce the following equation:

$$lnln\{1/(1-P_f)\} = wln\sigma - wln\sigma_0 + lnL \tag{12}$$

By plotting$lnln\{1/(1-P_f)\}$ versus $ln\sigma_0$ (this is commonly called as the weibull plot) a straight line of slope w, is obtained from which σ_0 (characteristic strength) can be found from the intercept with the x-axis.

P_f is obtained by ranking the data points in ascending order and using the following estimator:

$$P_f = \frac{j - 0.5}{n} \tag{13}$$

where,
n = number of data points
j = rank of the data point.

This estimator is used to give biased results for samples larger than 20.

Using Weibull distribution, values of strength obtained at one gauge length may be used to determine strength at another length for similar probabilities of failure using the following equation:

$$\sigma_{0(2)} = \sigma_{0(1)}(L1/L2)^{1/w} \tag{14}$$

where $\sigma_{0(1)}$is the strength of the fiber at length $L1$ and $\sigma_{0(2)}$ is the strength of a fiber of length $L2$. This is termed as a weak link scaling equation. Increasing the length is represented by a shift to the left on a Weibull plot.

Recently, Weibull distribution was also used for the analysis of tensile properties of natural fibers such as jute, cotton, hemp and flax. Xia et al., [90] have conducted studies on the breaking strength of jute fibers. Swapan et al.,[91] have studied the influence of chemicals on fiber structure and tensile properties of industrial hemp fibers. The physical and mechanical properties of cotton fibers were studied by Harzallah et al.,[92]

7 Experimental

7.1 Materials

Raw chicken feathers utilized for this project were procured from Wallace Corporation's Waitoa rendering plant. This rendering plant processes approximately 12 % of the North island's renderable material. Poultry material is separated out and processed through two lines, one for the feathers and one for poultry offal. Here, the feathers are rendered into feather meal.

The chemicals used in this project are as follows:

- 15 % Sodium hypochlorite (NaOCl) was used for decontamination (supplied by Univar) of the raw chicken feathers. 1M Sodium hydroxide (NaOH) was used for pH control during the decontamination process(supplied by Sigma Aldrich).
- To clean the decontaminated feathers, 30 % Hydrogen peroxide (H_2O_2) was used(supplied by Univar). It was diluted to 0.15 % and 0.25 % for the cleaning trails.
- The solvent used for soxhlet extraction was n-Hexane(supplied by Univar).

7.2 Equipments

The equipments used in the project are described below:

7.2.1 Lamort pulper

Feathers were decontaminated using a 30 L Lamort pulper shown in Fig.13. A flat disc agitator was used to prevent the entangling of feathers around the agitator.

Figure 13: Lamort pulper

7.2.2 Boltac mixer

A 6-unit Boltac mixer (shown in Fig.14) was used to conduct the cleaning trails of chicken feathers. Each unit had a working volume of 1 L and sirring speed up to 100 rpm. The experiments were conducted without temperature control.

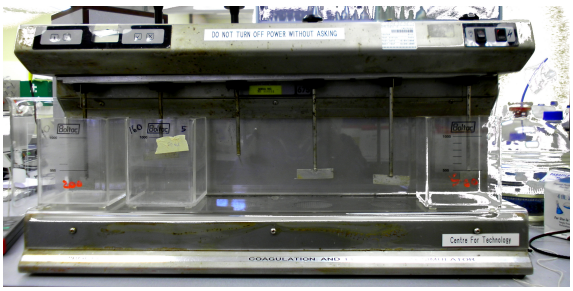


Figure 14: Boltac mixer

7.3 Soxhlet extraction apparatus

Extraction of soluble impurities from the feather samples were carried out using soxhlet extraction apparatus. A diagrammatic representation of the soxhlet extraction apparatus is shown in Fig.15.The components of this apparatus were: a soxhlet extractor, a water cooled condenser and a round bottom flask of 250 ml capacity. A cellulose extraction thimble of dimensions 33 x 100 mm was used to hold the samples inside the soxhlet extractor.

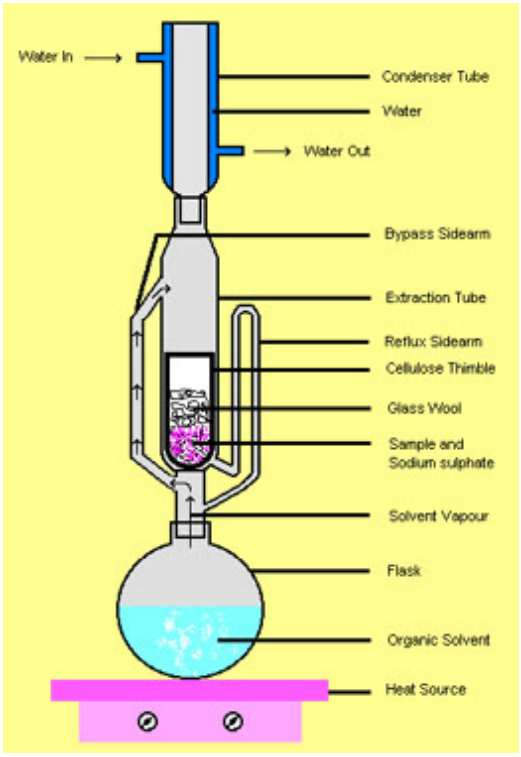


Figure 15: Representation of soxhlet extraction apparatus

The entire apparatus was clamped above a heating element (Thermo scientific).

7.3.1 Instron universal testing machine

Instron 33R4204 universal testing machine was used for single fiber tensile testing and a hot wire cutter was used to cut the supporting side of mounting cardboards. Tensile tesing of the fibers was carried out at a cross-head speed of 0.5mm/min using a 10N load cell. It is shown in the Fig.16 given below.

Figure 16: Instron universal testing machine

7.3.2 Image analysis

Optical microscope

An optical light microscope , Olympus BX60F5, fitted with a Nikon camera (Digital sight DS-U1) was used for image analysis of the feathers after treatment and was also used to measure fiber diameter for single fiber tensile testing. The samples were photographed at 3 magnifications (5X, 10X and 20X).

Scanning electron microscope

A Hitachi S4100 Field Emission Scanning Electron Microscope (FE-SEM) was used to examine the effect of cleaning on the surface of feather samples and also to analyze individual fibers obtained from the feathers. The specimens were coated with platinum and examined at 5Kv accelerating voltage. Magnifications used were 350x, 800x and 1300x.

7.3.3 Contherm air forced oven

After the finishing decontamination, cleaning and soxhlet extraction , the feathers were dried to constant mass in a contherm air-forced oven at 70^{o}C. The feathers were fluffed up once a while to assist in quicker evaporation of moisture.

7.3.4 Weighing balance

An ATRAX excell weighing balance was used to measure the weight of feather samples before and after processes like decontamination, cleaning and soxhlet extraction.

7.4 Experimental design

7.4.1 Kinetics experiments

The rates of impurity extraction of H_2O_2 from decontaminated chicken feather samples were determined by these experiments. The quantity of n-hexane extractable content remaining in the treated feather samples were also measured. In this experiment, the following points were considered:

- Mass of the decontaminated feather samples was kept constant (10 g).
- Mass of solvent was also kept constant (500 ml). H_2O_2 concentration was varied(0.15 % and 0.25 %).
- Time for leaching was varied (5,10,20,30,45 and 60 minutes).
- Number of cleaning stages was varied (1, 2 and 3).

7.4.2 Equilibrium experiments

The effect of cleaning on varied quantity of decontaminated feather samples was tested through this experiment. In this experiment, the following points were considered:

- The quantity and concentration of the solvent (H_2O_2) was kept constant (500ml and 0.25 % respectively).
- Time of leaching was fixed for 10 minutes.
- The quantity of decontaminated feather samples was varied. It ranged from 4 g to 17 g.

7.4.3 Single fiber tensile testing

This experiment was conducted to know the effect of H_2O_2usage for different time intervals on the mechanical strength of the fibers.

- Fibers treated with 0.25 % H_2O_2 were used for Tensile testing.

- The tensile test of fibers treated for 10 minutes (1 stage & 3 stage cleaning) and fibers treated for 60 minutes (1 stage & 3 stage cleaning) were compared. Fibers from decontaminated feathers were used as a control.
- Weibull analysis was done to predict the failure of the fibers.

7.5 Methods

7.5.1 Decontamination

Raw chicken feathers obtained from the rendering plant was collected in 10 L air-tight plastic buckets and kept in cold-storage room until decontamination. Raw feathers were decontaminated within 1 day.

The Lamort pulper was filled with 25 L water and 2.5 kg raw feathers. The pH of the suspension was tested using Litmus paper. To adjust the pH at 10.0, 1M NaOH was added. After the pH stabilized, 250 ml of 15 % NaOCl was added. The suspension was agitated at 10 Hz for a duration of 30 minutes. The liquid phase was drained over a 1mm mesh filter. The decontamination step was repeated once again. The decontaminated feathers were rinsed in 25 L water to remove residual NaOCl. The wet feathers were dried to constant mass in a contherm air-forced oven at 70^{o}C. This process is summarized in Fig.17.

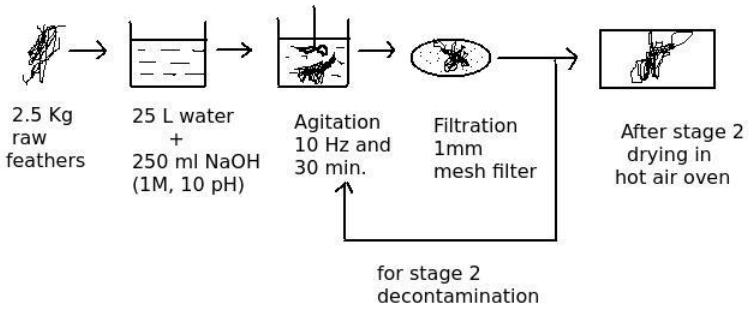


Figure 17: Representation of decontamination process

The feathers were fluffed up once a while to assist in quicker evaporation of moisture. Dried feathers were packed in zip-lock plastic bags and labeled.

7.5.2 Cleaning

10 grams of decontaminated feathers were agitated in the Boltac mixer at 60 rpm in 500 ml of hydrogen peroxide. The experiment was carried out to understand the kinetics of the cleaning process of decontaminated feathers.

Six different durations were considered for the cleaning process. These were, 5 minutes, 10 minutes, 20 minutes, 30 minutes, 45 minutes and 60 minutes.

One stage, two stage and three stage cleaning were carried out separately for each of the above mentioned duration of cleaning.

Hydrogen peroxide concentrations of 0.15 % and 0.25 % were used for cleaning process. The feather samples were rinsed in 500 ml of water after every stage of cleaning to remove residual H_2O_2. The process is summarized in the Fig.18.

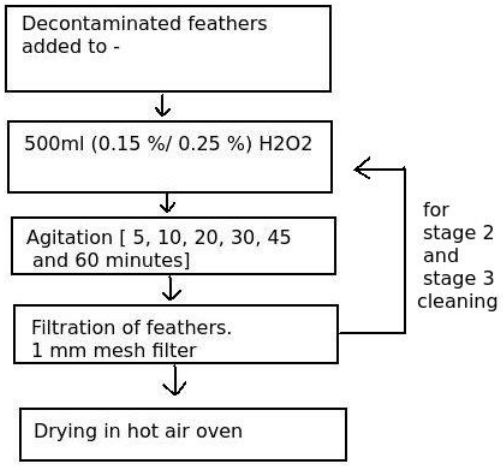


Figure 18: Flowchart of cleaning process

The wet feathers were dried to constant mass in a contherm air-forced oven at 70°C. The feathers were fluffed up once a while to assist in quicker evaporation of moisture. The dried feathers were packed in zip-lock plastic bags and labeled. The weights of the cleaned/dried feathers were measured and the difference in weight before and after cleaning was calculated.

7.5.3 Analysis of soluble impurities

Soxhlet extraction was used to analyze the content of soluble impurities from every sample of cleaned feathers. The solvent used in this process was n-Hexane. The extraction was carried out overnight and the feather samples were collected from the soxhlet extractor on the next day. The feathers were dried to constant mass in a contherm air-forced oven at 70°C. The dried feathers were packed in zip-lock plastic bags and labeled. Their weights were measured and the difference in weights before and after soxhlet extraction were measured. The solvent n-Hexane was recovered through the process of distillation.

7.5.4 Equilibrium experiments

Equilibrium experiments were carried out in 6-unit Boltac mixer at 60 rpm. 500 ml of 0.25 % Hydrogen peroxide was used as a solvent. The quantity of the chicken feathers tested ranged from 4 grams to 20 grams. A duration of 10 minutes was used for the cleaning and using only one stage. The solvents were filtered using a 1 mm mesh hand held filter. The feather samples were then dried to constant mass in a contherm air-forced dryer at 70°C . The feather samples were fluffed once in a while to assist in quicker evaporation of moisture. After the samples were dried, they were packed in zip-lock plastic bags and labeled. Then, the samples were subjected to soxhlet extraction to analyze the quantity of soluble impurities in each of the cleaned feather samples.

7.5.5 Separation of fibers from feathers

Separation of fibers from feathers (Barbs were removed from the rachis of the feathers) of each sample was done manually using scissors. The fibers were packed in Zip-lock plastic bags and labeled. The fibers are shown in the Fig.19.

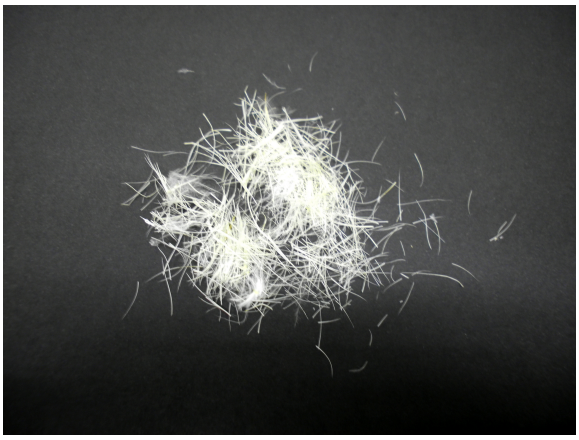


Figure 19: Fibers after separation from feathers

7.5.6 Single fiber tensile testing

The tensile strength of single feather fibers were measured according to ASTM D3379-75 standard test method for tensile strength and Young's modulus for single filament materials. The fibers were separated manually by hand and the single fibers were attached to cardboard mounting cards using a Poly Vinyl Acetate glue. A gauge length of 5mm was selected. The diameter of the fibers were measured at three different points along each fiber using an optical microscope. The average diameter of each fiber was calculated and it was considered as the fiber's diameter. The fibers mounted on the cardboards were placed in the grips of an Instron universal testing machine and a hot wire cutter was used to cut the supporting side of the mounting cardboards. Tensile testing of the fibers was carried out at a cross-head speed of 0.5 mm/min using a 10N load cell. 24 specimens were used for each sample. The shape of the fibers were assumed to be cylindrical. Bluehill 2 software was used for the calculation of Tensile strength and Young's modulus.

8 Results and Discussions

8.1 Kinetics experiments

8.1.1 0.15 % Hydrogen Peroxide cleaning

500 ml of the solvent (0.15% hydrogen peroxide) was used to leach impurites from 10 grams of feathers. This suspension was agitated in a six-unit Boltac mixer at 60 rpm for different time intervals. The feathers were dried and their weights were measured to estimate the percentage of impurities leached during this process. The weight loss percentage was plotted against time intervals of treatment (in minutes) as shown in Figure 20.

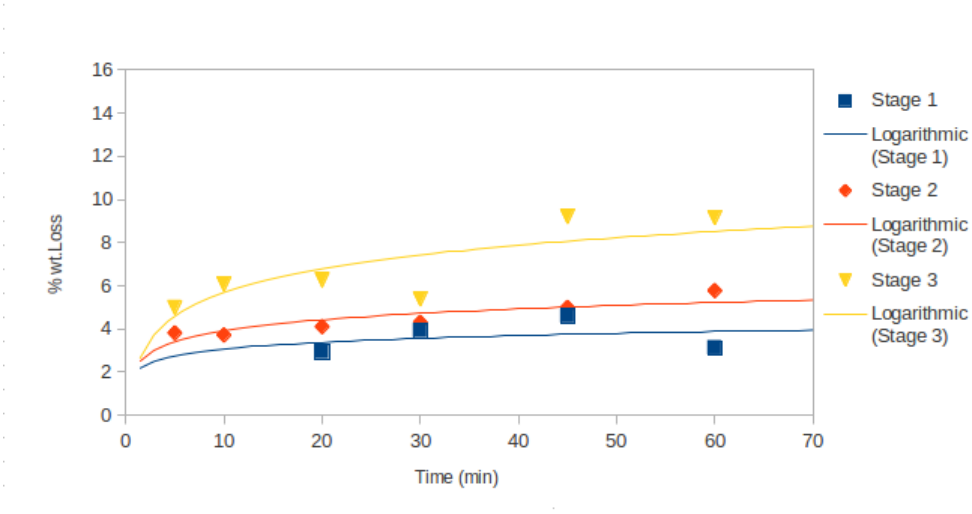


Figure 20: Leaching of impurities from feather samples treated with 0.15 % H_2O_2.

One, two and three stage leaching were carried out separately and the results are included in Figure 8.1. It can be seen from this figure that the average impurity removal per stage ranges from 4 to 6 %. Majority of the mass loss occured within the first 10 minutes. Weight loss results after 10 minutes remained mostly unchanged.

8.1.2 Impurity evaluation

To further clarify the extent of impurity removal, the hexane extractable content of the cleaned feathers were evaluated using soxhlet extraction. The impurities were expressed as n-Hexane extractable content (%) and were plotted against each sample's treatment time as shown in Figure 21.

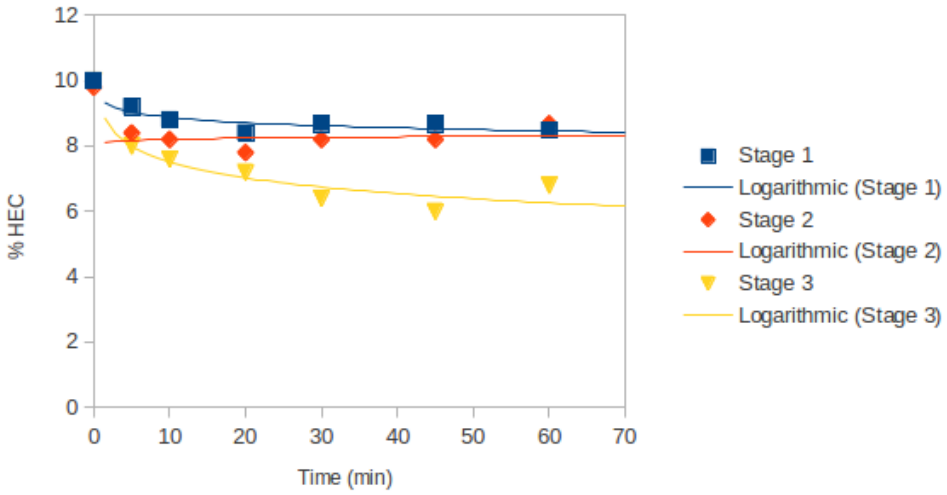


Figure 21: Hexane extractable content from feather samples treated with 0.15 % H_2O_2.

In the Fig.21, the zero minute values correspond to the hexane extractable content of decontaminated feathers (these are unwashed feathers). They had the highest HEC content of 10 %. It can be seen from the graph that there is a gradual reduction of HEC content as more stages were used. It can also be seen that there is no significant reduction in HEC after 10 minutes. This would suggest that 10 minute cleaning is sufficient for cleaning feathers when using 0.15% H_2O_2 solutions.

8.1.3 0.25 % Hydrogen peroxide cleaning

500 ml of the solvent (0.25% hydrogen peroxide) was used to leach out impurites from 10 grams of feathers. Weight loss percentage was plotted against time intervals of treatment (in minutes). one, two and three stage leaching were carried out separately and the results are included in Figure 22.

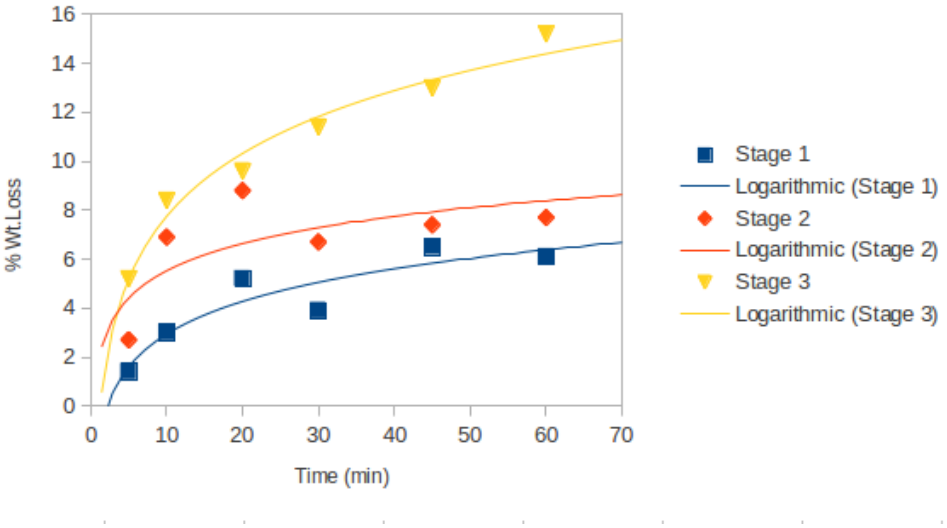


Figure 22: Leaching of impurities from feather samples treated with 0.15 % H_2O_2

From Fig.22 it can be seen that using a stronger solution did not significantly change the % weight loss. However, equilibrium was not reached until about 20 minutes. Also, considering all 3 stages, a plateau in mass loss was not observed with the 60 minutes samples used for testing.

8.1.4 Impurity evaluation

The analysis of soluble impurities in the feather samples cleaned with 0.25 % H_2O_2 were carried out using Soxhlet extraction. Hexane extractable content (%) was plotted against each sample's treatment time. It is shown in Figure 23.

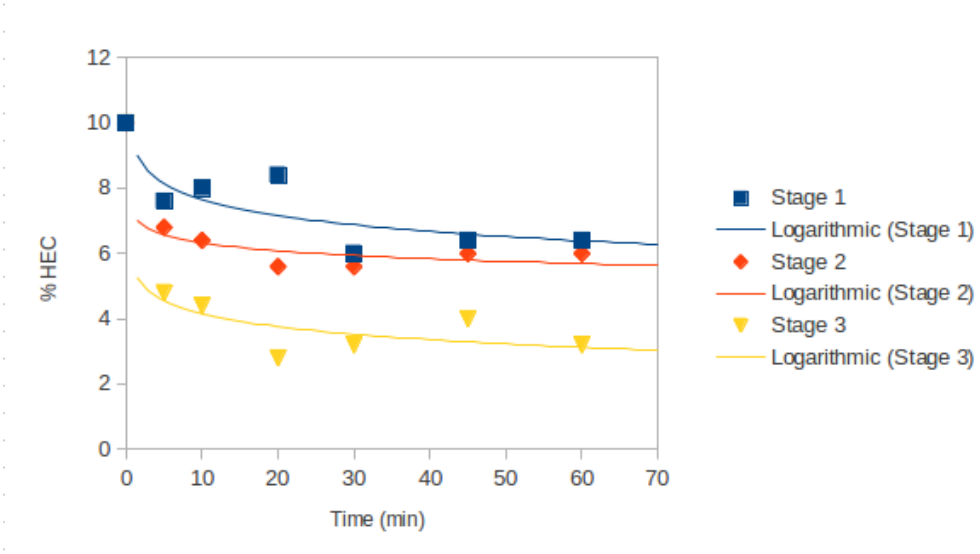


Figure 23: Hexane extractable content from feather samples treated with 0.25 % H_2O_2.

As before, the zero minute values correspond to the decontaminated samples. It can be seen from the graph that there is a gradual reduction of HEC content as the stages increase. A sharp drop of values is clearly observed for 5 minute and 10 minute treated samples after which the HEC % plateaus. The values of HEC % obtained in this experiment is lower than that of the 0.15 % H_2O_2samples. Also, there is a clear difference in values obtained from the three stages of cleaning. From these results, it can be concluded that 0.25 % H_2O_2treatment for feather samples is more effective than 0.15 % H_2O_2treatment for feather samples. Hydrogen peroxide treatment of more than 0.25 % was not tried, as previous studies conducted by Tseng,F-C.J[5]; revealed that the cleaning efficiency levelled off after 0.25 % hydrogen peroxide treatment of feathers.

From the above discussed results, it can be infered that 10 minutes treatment of feathers in 0.25 % $H_2O_2$is effective for removing impurities. There is no significant increase in the efficiency of leaching after 10 minutes. Also, a 3 stage process is better at leaching of soluble impurities from the feather samples. However, there are variations in the values obtained. The variations

are due to the biological nature of the chicken feathers. The feathers are harvested from many birds which are of diverse age groups and differ in amounts of Lipids and preen oils in their feathers.

8.2 Equilibrium experiments

Different amounts of decontaminated feather samples were leached once for 10 min in 500 ml of solvent (H_2O_2). They were filtered and dried to constant mass and the difference in their weights were measured. The Wt.loss (gram lipids) for each sample per 500 ml was noted. These values (gram lipids) were later converted to milligram lipids per ml of the solvent. The impurities extracted by the solvent for different quantities of feather samples were calculated. The results are shown in Fig.24

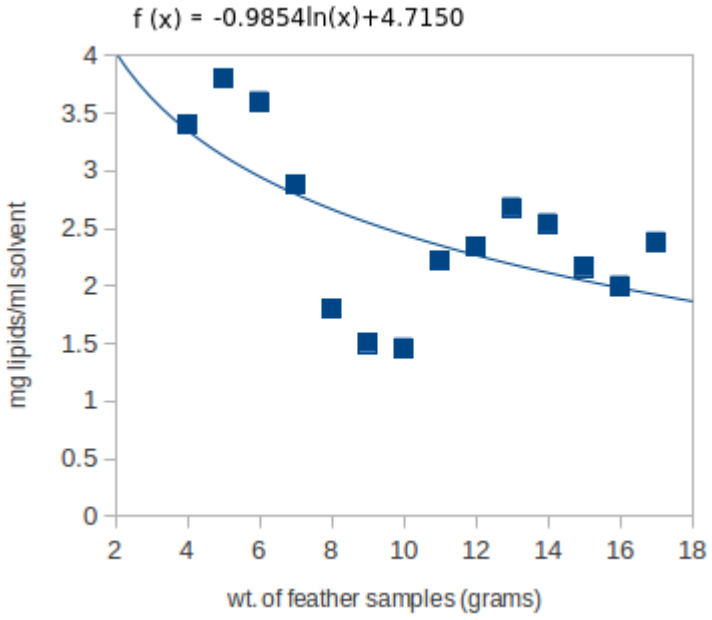


Figure 24: Quantity of lipids removed from one stage leaching for 10 minutes.

It can be seen that the quantity of the lipids extracted by the solvent decreased as the ratio of solid feed to solvent increased. Due to the biological nature of the feathers, the results were highly variable and a clear trend was absent. Processing could also have affected this, such as non-ideal mixing. Also,

any large quantities of feather could have required larger equilibrium.

To know the quantity of impurities remaining on the feather samples after treatment, soxhlet extraction of the leached samples was conducted. From each sample 2.5 g of feathers were used for the soxhlet extraction process. The weight loss was calculated after soxhlet extraction and was noted as gram lipids/2.5 g feathers.The results are shown in the Fig.25.

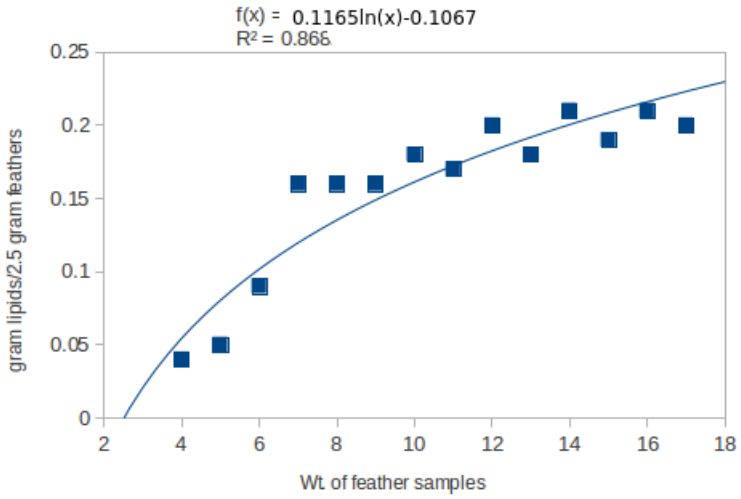


Figure 25: Quantity of Lipids remaining on the feather samples after leaching.

It can be seen from the Fig.7.6 that the quantity of lipids extracted by soxhlet extraction increased as the ratio of solid feed to solvent increased. This showed that the treated samples whose solid feed to solvent ratio was high had higher amount of lipids on its surface. These values supported earlier observations from Fig.24.

The values obtained are highly variable. A trend line was fitted and to make the calculations easier, the values on the trend line were considered. It is shown in Fig.26.

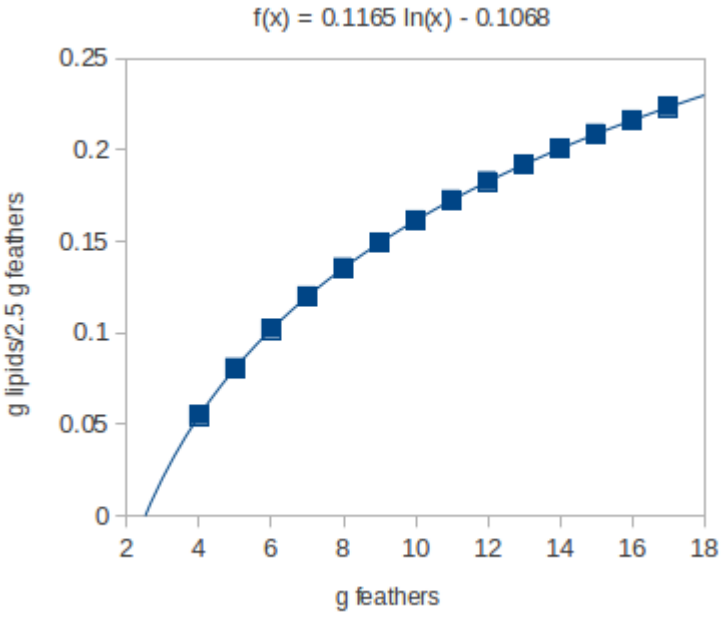


Figure 26: Quantity of lipids remaining on the feathers after leaching (calculated).

From this data, total lipids in the feather samples (i.e. lipids left on feathers after leaching with H_2O_2) were calculated. Then, the weight of feathers excluding lipid weights was calculated. This was based on the Soxhlet extractions with decontaminated feathers which were never washed with H_2O_2, they had a lipid content of 10%. From these above mentioned data (total lipids in feather samples and feather weight) the values of mg lipids/g feather was calculated. From these values, mg lipids/g solvent can be calculated.This is needed for plotting an equilibrium graph (mg lipids/g feathers vs mg lipids/g solvent). The discussion of mass balance in all these reactions will be helpful in plotting of the equilibrium point.

Mass balance The components involved in leaching are analyzed and represented as a mass-balance as shown in Fig.27

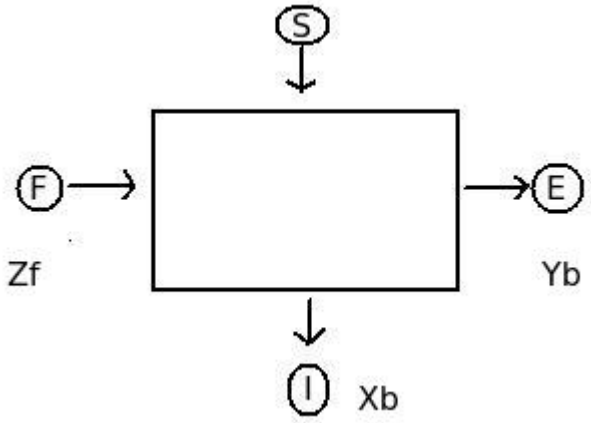


Figure 27: Mass balance in leaching of feathers.

Here, in the Fig. 27 the terms represent the following.
F= Fiber dry wt. (Dry fiber + lipids). Units are grams.
I= Dry clean fibers obtained. Units are gram/hr.
S= solvent (grams)
E= solvent/hr
Z_f is the lipid content in 'F' (units are mg lipids/g fiber).
Y_b is the lipid content in the solvent (units are mg lipids/ g solvent).
X_b is the lipid content after cleaning.
The amount of clean fibers is always constant.
so, I=F.
The amount of solvent exiting the system is constant.
so,E=S.
This relationship is given by the equation:

$$Z_f.F = Y_b.E + X_b.I \tag{15}$$

This equation can be further simplified as:

$$Y_b = -\frac{F}{S}X_b + \frac{F}{S}.Z_f \tag{16}$$

This is of the form $Y = mx + c$. Then, $\frac{F}{S}$ is the slope and $\frac{F}{S}.Z_f$ is the y intercept. Which implies that when $Y_b = 0$ then,

$X_b = Z_f$.

Substituting the experimental values in the above equations, the following values shown in Table 4 were obtained:

Table 4: Equilibrium values

Variables	Values
solid feed	10 g
Z_f	111.11 mg lipid/g fibre
Fibre content (F)	9 g fibre
Solvent (S)	500 g
F/S	0.018
F/S.Z_f	1.99

These values were plotted with mg lipids/g feathers vs mg lipids/g solvent to obtain the equilibrium graph shown in Fig.28 :

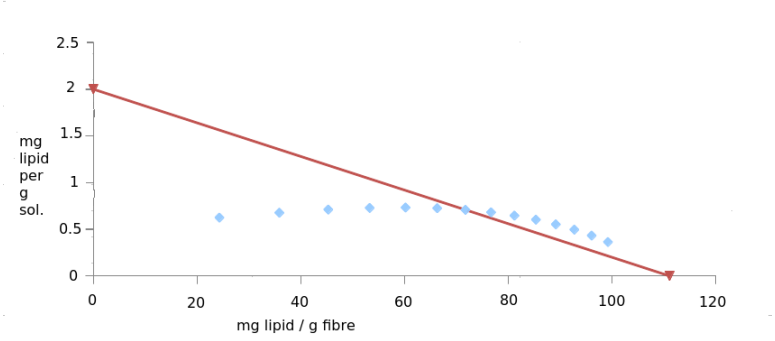


Figure 28: Equilibrium graph

It can be seen from Fig.28 and Table 4 that Z_f= 111.11 and the value of Y_b is 2. This operating line intersects the equilibrium line at 70 mg lipid/g fibre. This means that the lipids from the feather are not completely removed and still need further cleaning. It can be seen from the graph that two more washing stages would remove the lipid contents from the feather fibres.

The Chicken feathers used in the experiments are obtained from a very heterogenous population and the lipid content in the feathers vary to a great extent. Conducting rigorous experiments with very high samples may yield statistically

significant results in this regard. This data could be used for modeling of counter current extraction system for cleaning chicken feather fibers.

8.3 Effect of cleaning on morphology of feathers

Sodium hypochlorite and hydrogen peroxide were used for decontamination and cleaning. These chemical compounds are very widely used bleaching agents. The raw feathers obtained from meat processing plant are yellow in colour, clumpy and greasy. During the process of decontamination and cleaning, they undergo gradual colour change from yellow to white. There is a change in colour, texture and structure of the feathers after processing. By observing the morphology of the feathers, analysis on the extent of cleaning and the effect of cleaning on feathers can be known. In the following sub-sections, images of treated feather samples are shown and they are discussed in the sub-section 8.3.6.

8.3.1 Decontaminated feathers

The raw feathers from the meat processing plant are first decontaminated and dried. Below is an image of decontaminated feathers which are dried and packed.

Figure 29: Decontaminated feathers.

The decontaminated feathers look pale yellow to yellowish white in colour. There are characteristic yellow to dark yellow patches at the tips and base of the feathers. This indicates that the cleaning is not complete. The feathers mostly stick to one another. However, the decontaminated feathers do not smell bad

like the raw feathers. It is free from disease causing micro-organisms and is packed and can be stored for a long time.

8.3.2 0.15% H_2O_2cleaned feathers

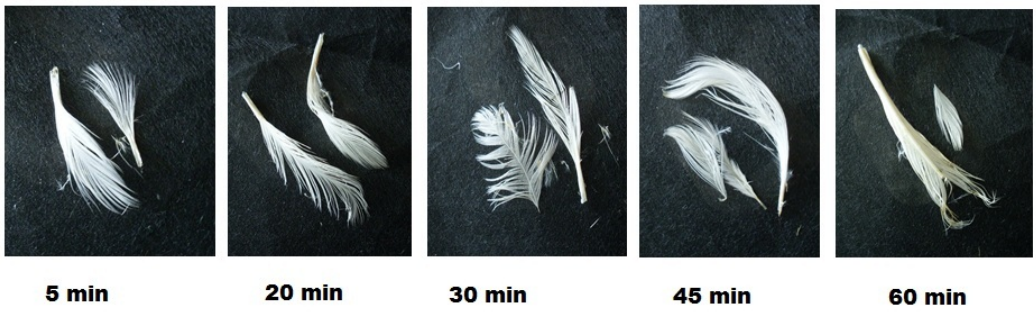


Figure 30: 0.15 % H_2O_2 stage 1 cleaned feathers.

Figure 31: 0.15 % H_2O_2 stage 2 cleaned feathers.

Figure 32: 0.15 % H_2O_2 stage 3 cleaned feathers.

8.3.3 0.25 % H_2O_2 cleaned feathers

Figure 33: 0.25 % H_2O_2 stage 1 cleaned feathers.

Figure 34: 0.25 % H_2O_2 stage 2 cleaned feathers.

Figure 35: 0.25 % H_2O_2 stage 3 cleaned feathers.

8.3.4 Microscopic morphology

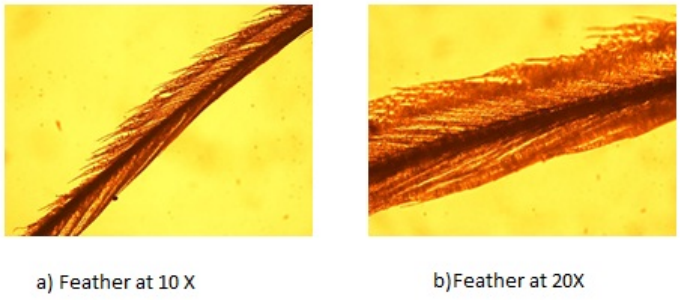


Figure 36: Surface of decontaminated feathers.

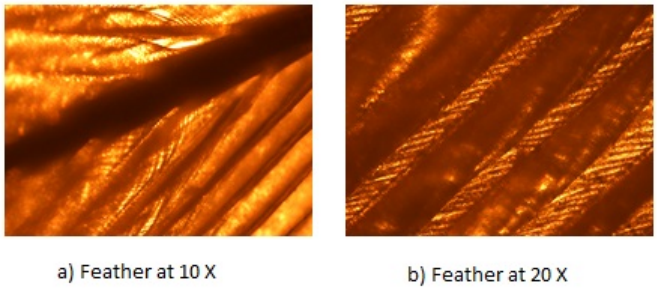
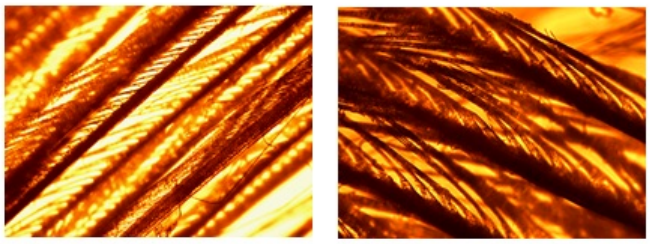
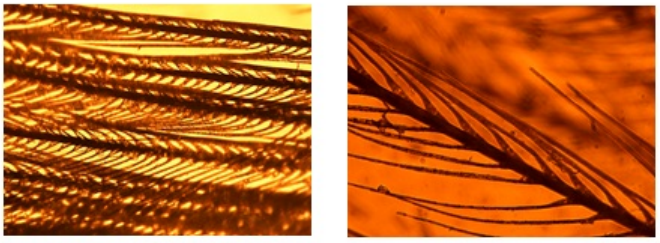


Figure 37: 10 minutes stage 1



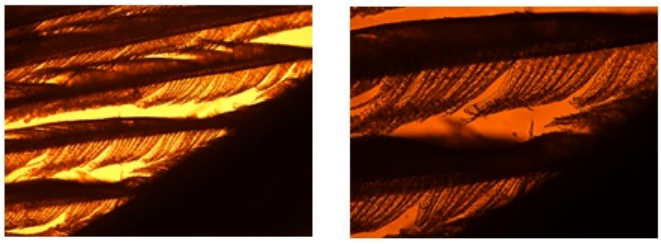
a) Feather at 10 X b) Feather at 20 X

Figure 38: 10 minutes stage 2



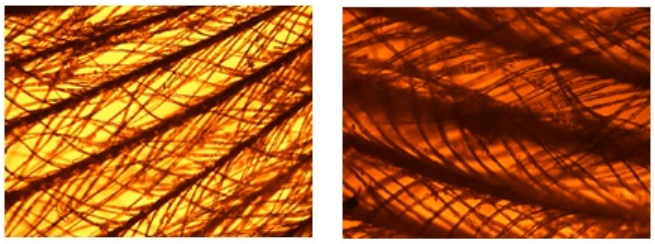
a) Feather at 10 X b) Feather at 20 X

Figure 39: 10 minutes stage 3.



a) Feathers at 10 X b) Feathers at 20 X

Figure 40: 60 minutes stage 1



a) Feather at 10 X b) Feather at 20 X

Figure 41: 60 minutes stage 2

a) Feather at 10 X b) Feather at 20 X

Figure 42: 60 minutes stage 3

8.3.5 Electron microscope images

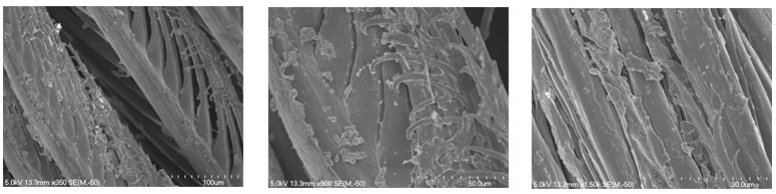


Figure 43: Decontaminated feather sample

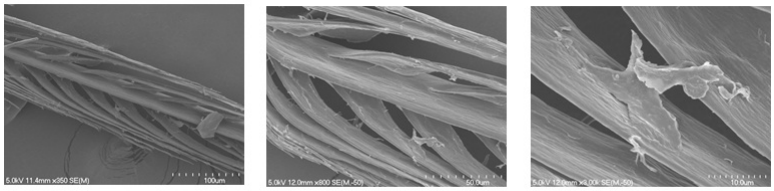


Figure 44: 10 minutes, stage 1.

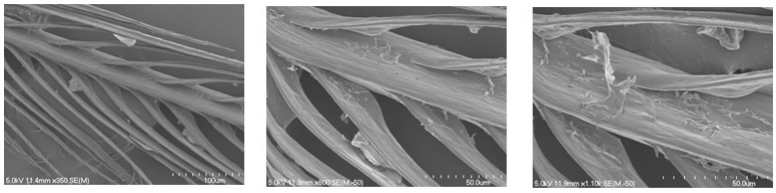


Figure 45: 10 minutes 2 stage cleaned feather sample.

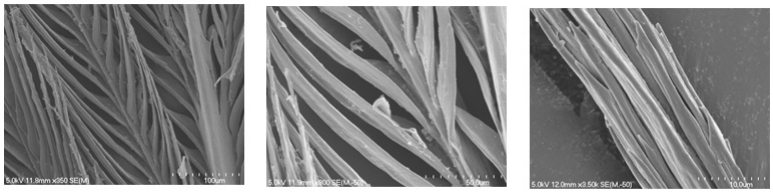


Figure 46: 10 minutes 3 stage cleaned feather sample.

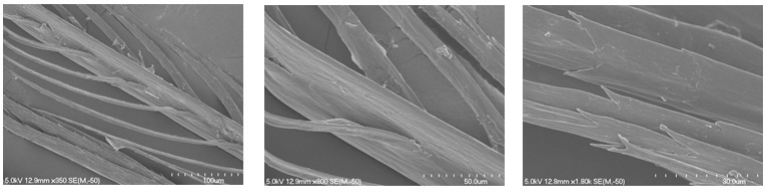


Figure 47: 60 minutes 1 stage cleaned feather sample.

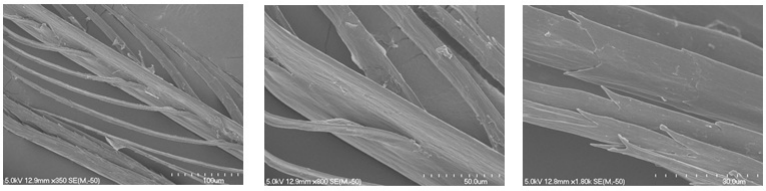


Figure 48: 60 minutes 2 stage cleaned feather sample.

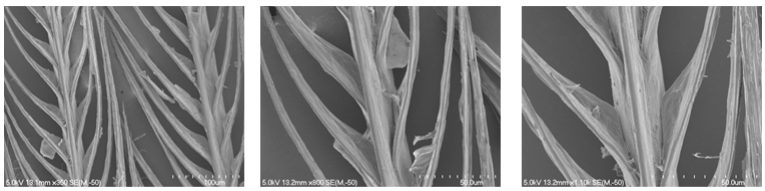


Figure 49: 60 minutes 3 stage cleaned feather sample.

8.3.6 Discussion on surface morphology of treated feathers

It can be observed from Fig.30 to Fig 35 that cleaning the decontaminated feathers with 0.15% and 0.25% H_2O_2 caused the feathers to become whiter with successive stages of washing. The feathers unfurl as the time and stages of washing increases. The texture of the feathers is smooth and fluffy after 3 stages of washing. No damage to the feather surface is seen. The Figures 36 to 42 were captured using an optical microscope (Olympus BX60F5), fitted with a Nikon camera (Digital sight DS-U1). It was observed that the decontaminated feather had clumpy appearance. Its fiber orientation was very dis-organized. Its barbules were connected very closely to one another and was clumpy. But, once the feathers were treated, uniformity in the orientation of the barbs and barbules were seen. There was increased spacing between the barbules as the duration and stages of the treatment increased.

A Hitachi S4100 Field Emission Scanning Electron Microscope (FE-SEM) was used to capture the images shown in Fig. 43 to 49. It can be observed from these images that the surface of decontaminated feathers had siginificant amount of impurities which blocked the gaps between the barbules which resulted in clumped feathers. The treated feathers showed gradual reduction in

the impurities as the duration and stages of treatment increased. No fiber damage was observed in any images.

8.4 Single fiber tensile testing

8.4.1 Single fiber morphology

The effect of hydrogen peroxide treatment on the mechanical properties of single fibers are discussed below. The barbs were cut off from the rachis of the feathers using scissors and they were packed and labeled. Single fibers representing each treatment were selected and their length and diameter were measured. The single fibers used for the tests ranged from 12 mm to 25 mm in length. Their diameters ranged from from 120 to 370 micrometers. Measurements were done using an optical light microscope (Olympus Bx 60 F5) fitted with a Nikon camera (Digital sight DS-U1). An example is shown below in Fig. 50.

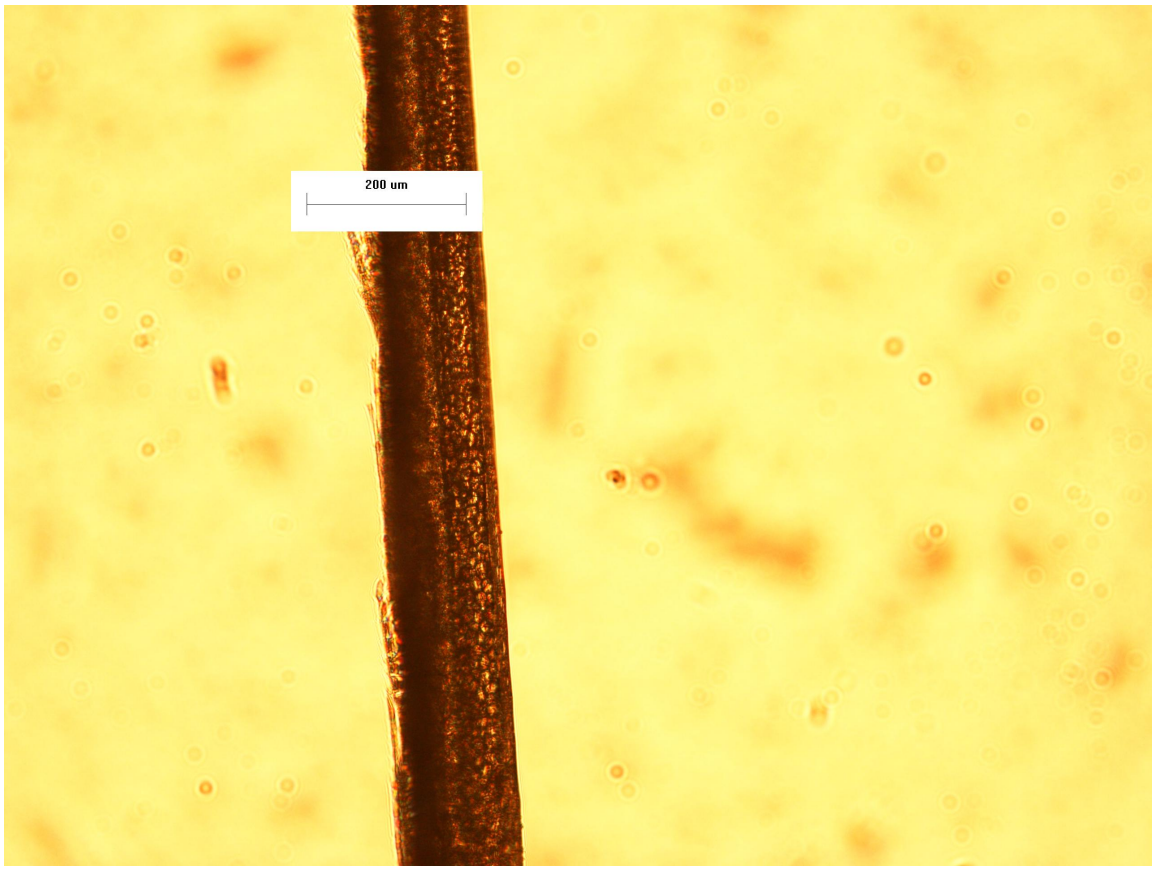


Figure 50: Fiber surface morphology

The diameter of the fibers is not constant throughout its length. The fiber is not perfectly cylindrical in shape. It has lot of irregularities along its length, like the protruding barbules. It can be clearly observed through electron microscope images, like Fig.51:

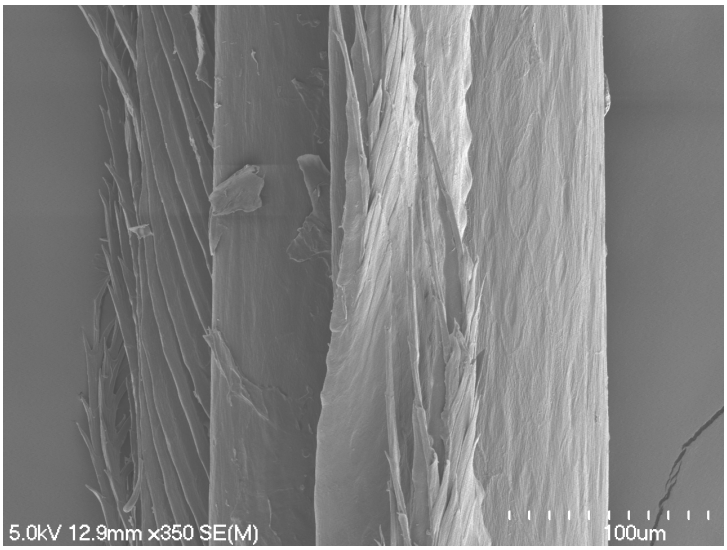


Figure 51: A close look at fiber surface morphology

8.4.2 Tensile testing

The single feather fibers were attached to cardboard mounting cards using polyvinyl acetate (PVA) glue. A gauge length of 5 mm was selected. Tensile testing of these single feather fibers were done according to ASTM D3379-75 standard test method. The feathers that were treated for 10 minutes (stage 1 and stage 3), 60 minutes (stage 1 and stage 3) and decontaminated ones were considered for tensile testing. 24 samples for each treatment were tested. Refer to raw data in appendix.

The tensile stress values of the fibers were plotted against diameter, to observe their relationship. The 10 minutes stage 1 samples are represented in Fig.52.

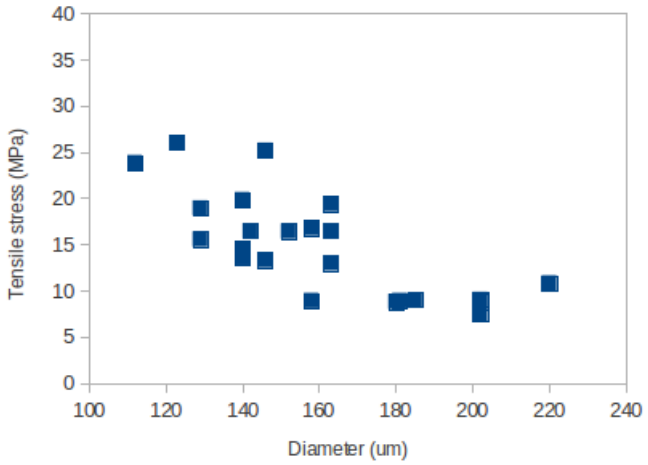


Figure 52: Tensile stress Vs. Diameter, 10 minutes, stage 1 fibres

It can be observed from the above figure that there seems to be an inverse relationship between diameter and tensile stress. The lesser the diameter, the more the tensile stress. The average tensile stress and average diameter of these samples were found to be 14.08 MPa and 155 μm respectively. (Average values are shown in Table)

The 10 minutes 3 stage samples are represented in Fig.53.

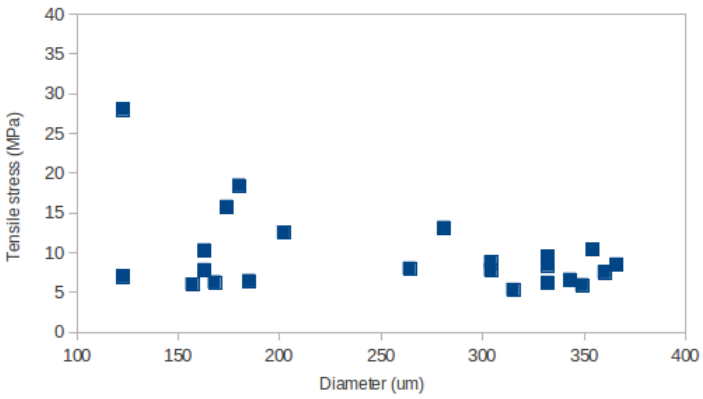


Figure 53: Tensile stress Vs. Diameter, 10 minutes, stage 3 fibres.

It can be observed that the fibers of this sample had a very wide range of

diameters. The tensile stress values are very low and seem to be inconsistent, highest being 28 MPa and many as low as 5 to 6 MPa. The average tensile stress value is 8.205 MPa and the average diameter is 272.5 μm.

In the 60 minutes washed samples (1 stage), shown in Fig.54, there is a clear trend of Tensile stress and diameter being inversely proportional. Most of the samples here, have stress values around 10-15 MPa and very few samples have extreme values. The average tensile stress and diameter values for these fiber samples are 12.83 MPa and 213 μm.

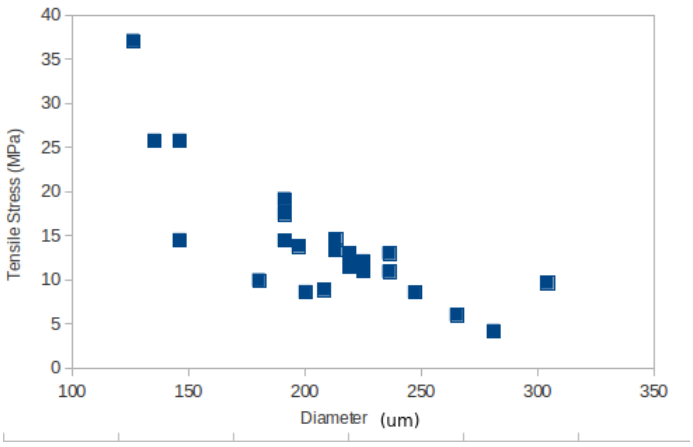


Figure 54: Tensile stress Vs. Diameter, 60 minutes , stage 1 fibres.

In the next sample, the 60 min. stage 3 cleaned fibers (as shown in Fig.55) have a consistent range of diameters and Tensile stress. There are very few extreme values. Their average tensile stress and diameter values are 16.105 MPa and 164.5 μm.

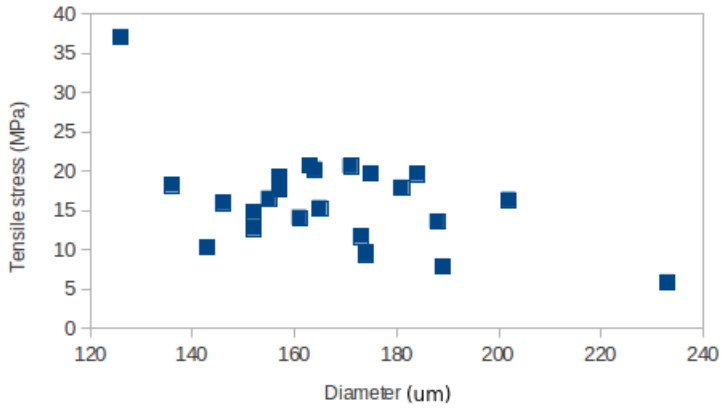


Figure 55: Tensile stress Vs. Diameter, 60 minutes , stage 3 fibres.

In Fig.56, the decontaminated fiber samples are represented. These samples too have a very wide range of fiber diameters. The samples having lesser diameters have more tensile strength. Their average tensile stress and diameter values are 15.63 MPa and 222.5 μm.

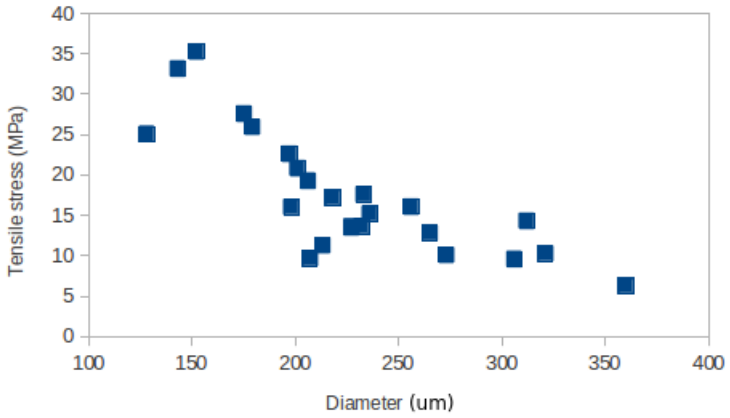


Figure 56: Tensile stress Vs. Diameter, Decontaminated fibres.

From the above produced graphs, it can be observed that the fibers having lesser diameters have better tensile stress than those having higher diameters.

It suggests an inverse relationship between diameter of the fiber and the tensile stress. The average values of Tensile strength and diameters of the tested chicken feather fibers are given in Table 5 below:

Table 5: Average values of Tensile stress and Diameters.

Sample	Tensile stress (MPa)	S.D	Diameter μm	S.D
10 min. S1	14.08	5.433	155	28.241
10 min. S3	8.45	13.778	272.5	84.848
60 min. S1	12.835	7.105	213	43.455
60 min. S3	16.105	6.191	164.5	22.569
Decontaminated	15.63	7.556	222.50	57.311

The values of the average tensile stress and Young's modulus alone does not give a clear picture on the distribution of fiber strengths.It is inaccurate in predicting the failure rate and wearing out of fibers, as it relies on mean values.

Weibull statistics can be used to model the strength of the fibers accurately. Through this method, a characteristic strength of the fiber (σ_0) for which the probability of failure is 63.2% is calculated.

8.4.3 Weibull statistics

As described in the literature review, a Weibull plot is obtained by plotting ln(ln(1/1-Pf)) Vs. ln (σ_0). The slope obtained through these plots gives a value 'W'. It describes the variability of failure strength of a fiber of length L. In these experiments, L=5mm (gauge length) which was maintained constant. The x-intercept obtained from the Weibull plot is the value of Ln(σ_0). From this, the value of σ_0,the characteristic strength of the fiber can be derived.

Weibull analysis was carried out for all the samples and its results are sequentially shown from Fig.57 to Fig.61. The values of W and (σ_0) are shown in Table 6.

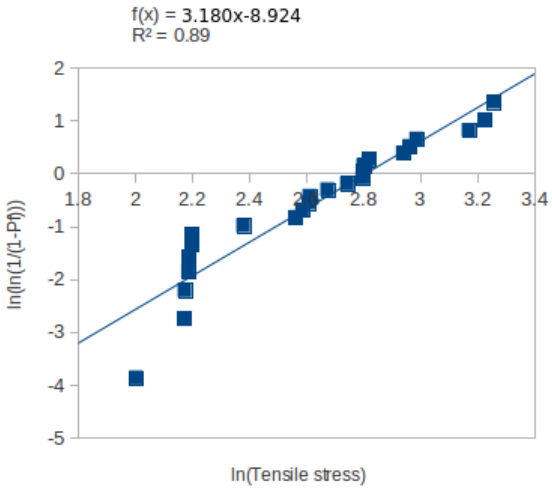


Figure 57: Weibull plot: 10 min. stage 1.

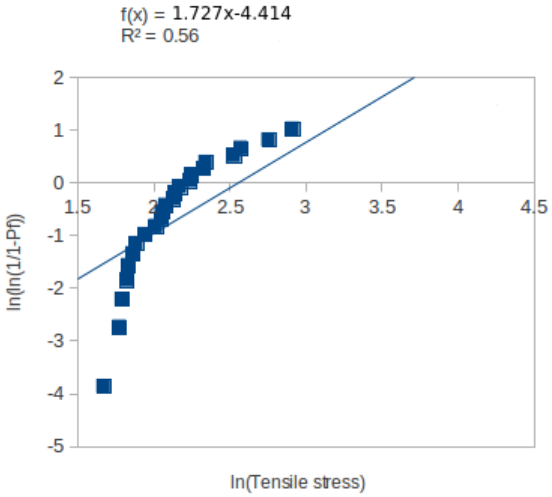


Figure 58: Weibull plot:10 min. stage 3

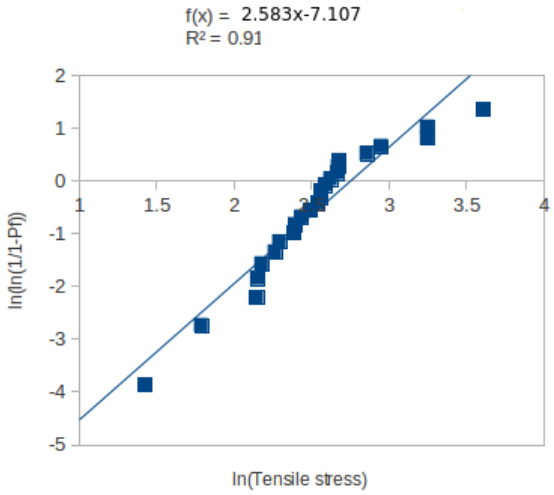


Figure 59: Weibull plot : 60 min. stage 1

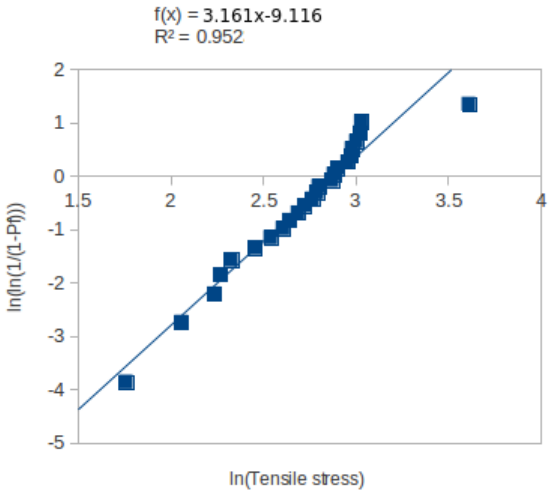


Figure 60: Weibull plot : 60 min.stage 3

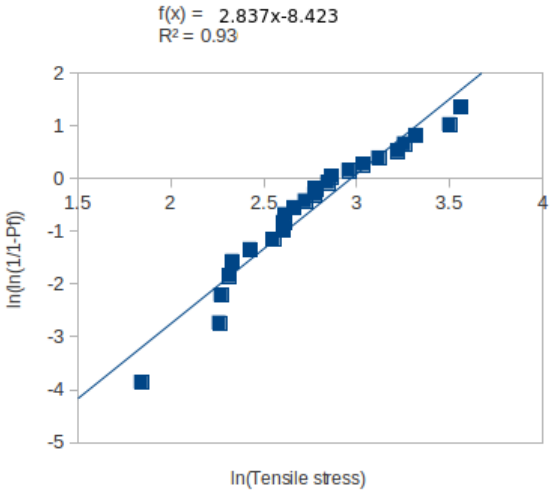


Figure 61: Weibull plot: decontaminated sample.

Table 6: Weibull analysis

Treatment	W	σ_0(MPa)
Decontaminated	2.83	19.5
10 min x 1	3.18	16.45
60 min x 1	2.58	15.64
10 min x 3	1.72	12.81
60 min x 3	3.16	17.86

From the Table 7.1 it can be seen that the fiber sample which was treated for 10 minutes 3 stages has a very low 'W' value. A low 'W' value indicates high variability in failure strength.

The expected order of failure strength of the fiber samples were:

Decontaminated>10 min (1stage)>60 min (1stage)>10 min (3 stage)>60 min (3 stage).

There was not much difference in the observed results.

The data suggests that tensile strength of the treated fibers decreases as the duration and stages of treatment increases. But, big difference in tensile strength was not observed. The treatments on the fibre did not not cause significant damage to it.

Chicken feather fibers are very small and difficult to do single fiber tests. Only lengthy fibers (12-25 mm) were considered for this test so that a gauge

length of at least 5 mm could be made use of. The tensile stress values of the fibers were very low, compared to those found in literatures. Considering this fact, the entire experiment was checked for errors.

The following Fig.62 highlights the error.

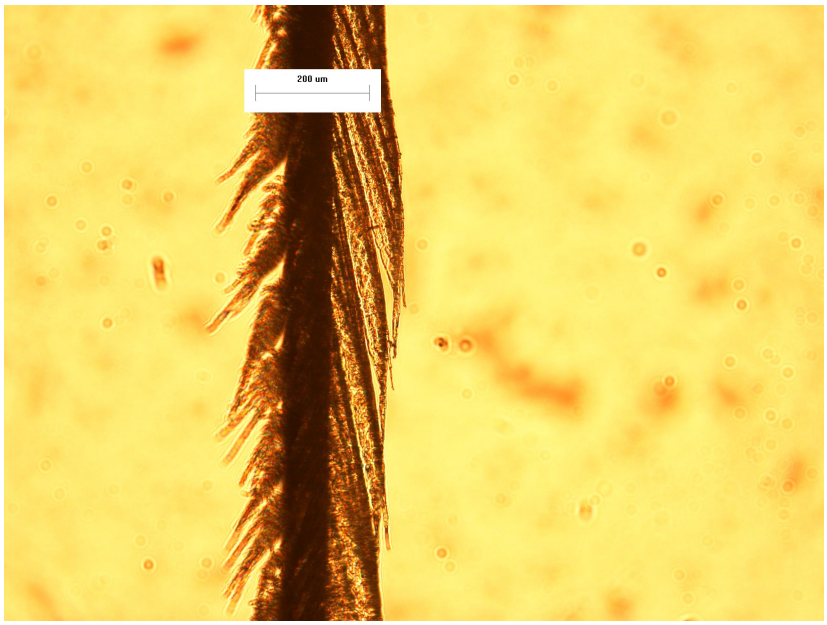


Figure 62: Misinterpreted diameter

From Fig. 69 it can be observed that the barbules of the fiber have also been included during the measurement of diameter of the fiber. The real fiber diameter is actually $\frac{1}{3}$ times lesser than the recorded diameter. Such a change could increase the tensile stress values by nearly 9 times. An example of change in Tensile stress values due to apparent and real diameter are shown in the Table 7.

Table 7: Examples of tensile stress corrections

Ap. diameter(μm)	True diameter(μm)	Ap.tensile stress(MPa)	True tensile stress(MPa)
127	50.8	15.52	41.67
146	73	25.19	178.85
112	61	23.84	69.55
185	74	9.01	105.55
180	90	8.8	144.5
129	86	18.93	145.68
140	70	13.59	81.63
202	101	7.42	193.17
140	46.66	14.52	38.73
140	70	13.64	81.9

The correction in diameter was done by analysis of the microscope images which were originally used for measuring the diameter. The values shown in table 7 belong to the 10 minutes 1 stage washed samples. These types of errors need to be corrected to obtain accurate values for modelling the strength of fibers.

9 Conclusions

The raw feathers collected from Wallace corporation were decontaminated using sodium hypochlorite. The decontaminated feathers contained about 10 % Hexane extractable content. The decontaminated feathers were cleaned using hydrogen peroxide. 0.25 % H_2O_2 was found to give cleaner feathers than 0.15 % H_2O_2.

Kinetics experiments revealed that 10 minutes cleaning for 3 stages is the most effective approach. The Hexane extractable content was brought down to 4% from 10 % after using this method. There was not much decrease in Hexane extractable content after this treatment.

The solid feed to solvent ratio used for the cleaning trial was 10g feather per 500 ml of solvent. A mass balance showing lipid transfer was designed. Analyzing the data from equilibrium experiments and applying mass balance, a graphical representation of equilibrium condition was presented. It suggests that 3 stages of cleaning is sufficient to remove majority of the lipids from the feather fibers, considering the 10 g feather per 500 ml solvent. This graph can be used to predict the lipid content removed by leaching under different conditions.

The H_2O_2 treated feathers looked fluffy in texture and white in color. The foul odor present in the raw feathers was also eliminated through cleaning process. Each stage of cleaning produced feathers of increasing whiteness and resulted in better spreading of the sub-structures of the feathers. The stickiness of the feathers was absent and it looked fit for use in composite materials.

Single fiber tensile test was done to study the effect of hydrogen peroxide treatment on the fibers. 24 specimens per sample were considered for the test. The decontaminated fibers had higher values of tensile strength than the treated fibers. It was found that treatment does affect the mechanical strength of the fibers, but not by a great extent. The treatment with H_2O_2 did not damage the fibers.

9.0.4 Recommendation

A few more tests focussing on the tensile strength of the fibers need to be conducted to know the accurate values. Conducting the leaching experiments

on a large scale to check the rate of lipid removal would be informative for standardizing the cleaning process of chicken feather fibers.

References

[1] Sabat.R.,Rajak.R.K.(2009).*Sliding wear behaviour of biowaste reinforced polymer composite* (Bachelor's thesis).National Institute of Technology, Rourkela, India.

[2] Askeland,D.R. (1996).*The science and engineering of materials.*Third S.I edition.UK:Chapman & Hall.

[3] *New Zealand poultry meat production.* Retrieved from www.pianz.org.nz on 25/07/2012.

[4] *Poultry feathers- what can they be used for?* Retrieved from www.ca.uky.edu/smallflocks/feather_production-html on 22-03-2012.

[5] Tseng,F.C.J., (2011). *Biofibre production from chicken feather*, Master's degree thesis. University of Waikato, Hamilton, New Zealand.

[6] Acda.M.N.(2010). *Waste chicken feather as reinforcement in cement bonded composites.* Philippine journal of science 139(2),161-166.

[7] Zhan.M., and Wool.R.P.(2011). *Mechanical properties of chicken feather fibres.* Journal of Polymer composites,32,6 Proquest Science journal.

[8] Strong,A.B.(n.d.).*History of composite materials-opportunities and necessities.* Retrieved from *https://strong.groups.et.byu.nbet/pages/articles/history.pdf* on 09-04-2012.

[9] Wanberg.J. (2009). *Composite materials fabrication handbook # 1*, Stillwater: Wolfgang Publications Inc.

[10] Why composites? Retrieved from https://compositecenter.org on 24-06-2012

[11] Pandey. P.C. (n.d). *Composite materials* (web based course) Department of civil Engineering, Indian Institute of Science, Bangalore, E-mail: pc pandey @civil. Iisc.ernet.in.

[12] Osorio.E.,Trujillo.E.,Vanvuure.A.W and Verpoest.I.(2011).*Morphological aspects & mechanical properties of single bamboo fibers and flexural characterization of bamboo/epoxy composites.* Journal of reinforced plastics and composites,30 (5), 396-408.

[13] *Kevlar-A technical guide.* Retrieved from www.dupont.com on 28-04-2012.

[14] Mohanty,A.K., Misra,M and Drzal,L.T.(2005). *Natural Fibers, Biopolymers and biocomposites.* Taylor and Francis group.

[15] Singha,A and Thakur,V.(2008).*Mechanical properties of Natural fibre reinforced polymer composites.* Journal of Polymer science,31 (5), 791-799.

[16] *Global education.* Retrieved from www.globaleducation.edna.edu.au/globaled/go/pid/3776 on 29-04-2012.

[17] *The University of Tennessee.* Retrieved from www.engr.utk.edu/mse on 29-04-2012.

[18] Cristaldi,G., Latteri,A., Recca,G and Cicala,G.(2010) *Composites based on natural fibre fabrics* In: Woven Fabric Engineering Ed: Dubrovski. Publisher:SCIYO.

[19] Bledzki,A and Gassan,J.(1999). *Composites reinforced with cellulose based fibers.* Journal of Polymer science,24 p.221- 274.

[20] Gomes,A., Goda,K., and Ohgi,J.(2004). *Effects of alkali treatments to reinforcement on tensile properties of curava*,In: green composites JSME Inte. J.Ser.A , 47 (4) ; pp: 541-546.

[21] Brouwer.W(2004).*Natural fiber composites in structural components:alternative application for sisal?* FAO report Y 1873 E.

[22] Murali.K., and Mohana.K.(2007).*Extraction and Tensile properties of natural fibers : wakka, date & Bamboo.* Journal of Composite struct,77: p. 288-295.

[23] Cao.y.,Shibatta,S., and Fukumoto.I.(2006). *Mechanical properties of biodegradable composite reinforced with bagasse fiber before & after alkali treatments.* Composites Part A,37: p.423-429.

[24] Okubo,K., and Fuji,T.(2002). *Ecocomposites using natural fibers & their mechanical properties* In- High performance structures & composites,Ed: Brebbia, C & de Wilde, W Southhampton : WIT press, p. 77-85.

[25] Cheung,H.Y., Ho.,M.P., Cardona,F., and Hui,D.(2009).*Natural Fiber–reinforced composites for bioengineering & Environmental engineering applications*,Composites: part B,40 p. 655-663.

[26] *Britannica*. Retrieved from www.britannica.com/EBchecked/topic/647753/wool on 30-04-2012.

[27] *Wool fiber*. Retrieved from www.wolframalpha.com/entities/common_materials/wool_fiber/w9/pa/00/ on 30-04-2012.

[28] Annadurai,V., Subramanyam.G., Gopalakrishne.URS., and Somashekar.R.(2001).*Structure–property relation in varieties of silk fibres*. Journal of applied polymer science,Vol. 79, p.1979-1985.

[29] *Te Ara*. Retrieved from www.teara.govt.nz/en/poultry-industry on 31/07/2012.

[30] *Global poultry trends (2012)- poultry increases its share of global meat production*. Retrieved from www.themeatsite.com on 28-04-2012.

[31] *Poultry processing*. Retrieved from www.britannica.com/EBchecked/topic/473031/poultryprocessing on 25/07/2012.

[32] Reddy,N and Yang, Y. (2010). *Light weight polypropylene composites reinforced with whole chicken feathers*. Journal of applied polymer science, 116(6), p.3668-3675.

[33] Reddy,N and Yang, Y. (2007). *Structure and Properties of chicken feather barbs as natural protein fibers*. Journal of Polymers & environment, 15 (2), 81-87.

[34] *The Cornell lab of ornithology*. Retrieved from www.birds.cornell.edu/allaboutbirds/studyingfeathers/feathers on 10-04-2012.

[35] Bock, W.J. (2004).*Birds in Encyclopaedia of life support systems*, Eds: Contrafatto,G and Minelli,A. Oxford, UK:EOLSS.

[36] Leeson.S and Walsh.T.(2004).*Feathering in commercial poultry.*World's poultry science journal, vol.60,42-51.

[37] Schmidt,W.F and Line M.J.(1996). *Physical and chemical structures of poultry feather fibre fractions in fiber process development* . Non-woven conference TAPPI proceedings (135-141).

[38] *Feather Bioloy*. Retrieved from http://askabiologist.asu.edu/explore/feather-biology on 10-04-2012.

[39] Kock,J.W. (2006).*Physical & mechanical properties of chicken feather materials.* Master's degree thesis. Georgia Institute of Technology, Georgia, United States.

[40] *Structure of keratin.* Retrieved from Chempaths.chemddl.org on 10-04-2012.

[41] Barone.J.R and Schmidt.W.F.(2005). *Polyethylene reinforced with keratin fibres obtained from chicken feathers.* Composite science & Technology, 65 , 173-181.

[42] Arai,K.M., Takahashi.R., Yokote,Y., and Akahane,K.(1983). *Aminoacid sequence of feather keratin from fowl.* European Journal of Biochem,132,501-507.

[43] Lucas,A.M and Stettenheim.(1972). *P.R Avian anatomy Integument*, parts I,II, III USDA Agri handbook # 362.

[44] Moran,E.T., Summers,J.D., and Slinger,S.J. (1996). *Keratin as a source of protein for the growing chick.* Poultry science ,45,1257- 1266

[45] Bitter.J A.(1963). *Study of Erosion phenomenon.* Part- I Wear 6, Vol. 6-Issue- I,5-21.

[46] McGovern.M.(2000). *Recycling poultry feathers: More bang for the cluck.* Environmental health perspectives, volume-108, no. 8, august.

[47] Bullions.T.A., Hoffman.D., Gillespie.R.A., Price-o-Brien,J., and Loos. A.C.(2006). *Contributions of feather fibres and various cellulose fibres to the mechanical properties of polypropylene matrix composites.* Composites science & Technology, 66, 102-114.

[48] Kar, P. and Mishra.M.(2004). *Use of Keratin fiber for separation of heavy metals from water.* Journal of Chemical Technology & Biotechnology, 79, 1313-1319.

[49] Cheung,Hoi-Yan., PoHo., Mei., Lau,Kintak., Cardona,F. and Hui,D.(2009). *Natural fibre reinforced composites for bioengineering & Environmental Engineering Applications.* J.Composites : Part B,40 p.655-663.

[50] Schmidt,W.F., and Jayasundara,S.(2004). *Microcrystalline avian keratin protein fibers*, chapter-4, In- Natural Fibers, plastics & composites, Eds: Wallenberger, F.T, Weston:N Kluwer Academic publishers.

[51] Gassner et al. *United States patent Patent no: 5705030.* Date of patent: Jan 6th, 1998.

[52] Schmidt, W.F.(1998). *Innovative feather utilization strategies.* Proceedings poultry waste management conference Springdate, Arkansas.

[53] Li, Hu-lin., Zhao,Y.Q., Lau;K.T. and Cheung,H.Y.(2010). *Chicken Feather Fiber/ polylactic acid green composites : Mechanical, Thermal & Biodegradable properties.* In: Green composites, properties,Design & Life. Eds: Willems,F., Moens,P.p119-139: Nova science publishers Inc.

[54] Bonser, R.H.C & Purslow, P.P The young' modulus of feather keratin Journal of Experimental Biology, (1995)198; p 1029-1033

[55] Hong,C.K. and Wool,R.P.(2005). *Development of bio based composite materials from soybean oil & keratin fibres.* Journal of applied Polmer Science,95, p. 1524-1538.

[56] Wool.R. and Hong.C.(2004). *Low dielectric constant materials from plant oils and chicken feathers.* United States patent application number 20040072976.

[57] Frazer,L.(2004).*Environmental health perspectives.* vol.112. no.10, July, pp: 564-567.

[58] Misra.M., Kar.P., Priyadarshan.G., and Licata.C.(2001). *Keratin protein nanofiber for removal of heavy metals & contaminants.* MRS symposium fall proceedings,702, 1-7.

[59] Rosa,G.de la., Reynel-Avila,H.E., Petriciolet.B., Cano-Rodriguez.I., Velasco-santos,C., Martinez-Hermandez,A.L.(2008). *Recycling poultry feathers for Pb removal from waste water : kinetic & equilibrium studies.* World academy of science, Engineering & technology, 47, p.p: 394-402.

[60] Wrzesniewska-Tosik.K., Wawro.D., Ratajska.M., Stplewski.W.(2007). *Novel biocomposites with feather keratin.* Fibres & Textiles in Eastern Europe Jan/Dec; vol.15, no.5-6 (64-65).

[61] George,B.R., Evazynajad.A., Bockaire.A., Mc Bride,H., Bunik,T., and Scutti.A.(2004). *Keratin based non-wovens for erosion control.* chapter-5, In- Natural fibers, Plastics & composites. Ed : Berger,F .T.W., Weston.N., : Kluwer Academic publishers.

[62] Ye,W., and Broughton,R.M.(2002). *Feather as a fiber source for non-woven insulation.* International non-wovens Journal, 8(1), 53-59.

[63] Senoz,E; Wool,R.P.(2011). *Hydrogen storage on pyrolyzed chicken feather fibres.* International journal of Hydrogen energy, 36, 7122-7127.

[64] Mokrejs.P et al.,(2011). *Processing poultry feathers into keratin hydrolysate through alkaline enzymatic hydrolysis.* Waste management and research,29 (Compendex) p.260-267.

[65] Mc Donald,N.,(2002). *Chemist puts chicken feathers to use in heavy duty products.* Federal Technology report, 14th april: McGraw Hill Inc.

[66] Stern,N.J., Line,J.E., and Chen,H.C.(2001). *Campylobacter.* In: Compendium of methods for the Microbiological Examination of foods, Ed : Downes,F.P. & Ito,K. American Public health Association : Washington, DC.P xxi, 676.

[67] AgResearch.(2008). *Testing meats & meat products for Pathogens Thermotolerant Campylobacter.* In: Meat industry Microbiological methods. AgResearch, Hamilton, p.7.

[68] AgResearch (2011).*Testing meats & meat products for Pathogens Salmonella.* In; Meat industry Microbiological methods. AgResearch, Hamilton,p.11.

[69] Kruchen,E.(1979). *Method of cleaning poultry feathers.* US4, 169,706 United States.

[70] Griffith, B.A.(2002). *Feather molding method and product.* US0, 169, 706 United States.

[71] *Laundry Bleaches.* Retrieved from www.jinke-chem.com/en/dispArticle.Asp?ID=1340 on 13-04-2012.

[72] Augurt,T.A., and Van Asten.J.(2000). *Sterilization techniques.* In: Kirk-othmer Encyclopaedia of chemical technology: John Wiley& Sons, Inc.

[73] Stoves,J.L.(1942) *The Reactivity of the cystine linkage in keratin fibers.* Part III The action of oxidizing agents (Published on 01 January 1942,http://pubs.rsc.org,DOI: 10.1039/TF9423800501).

[74] Howitt, F.O.(1955). *Wool, Silk and regenerated protein fibers chemistry.* Review of Textile progress, P 70-90.

[75] Popescu,C. and Hocker,H.(2007). *Hair-the most sophisticated biological composite material.* Chem.Soc.Rev., 36,p. 1282- 1291.

[76] Clegg,A.J., Loveman, E., Gospodarevskaya,E; Harris.P; Bird,A; Bryant.J. et al.,(2010). *The safety & effectiveness of different methods of earwax removal: a systematic review & economic evaluation.* Health Technol Assess,14 (28),1-192.

[77] Okuda,I; Bingham,B; Stoney,P; Hawke,M.(1991). *The organic composition of ear wax.* J.Otolaryngol. Jun; 20 (3): 212-5

[78] Jones, C.W.(1999). *Applications of Hydrogen peroxide and derivatives.* RSC clean technology, Monographs, Cambridge, U.K: The royal society of chemistry.

[79] Takeuchi,T.M., Pereira, C.G., Braga,M.EM., Marostica Jr,M.R., Leal,P.F., Meireies,M.A.A.(2009). *Low pressure solvent extraction (solid-liquid extraction, microwave assisted & ultrasound assisted) from condimentary plants.* In: Extracting Bioactive compounds for food products- Theory & Applications. Ed: Meireies,M.A.A,CRC press.

[80] Christi.J.G.(1993). *Transport processes & unit operations.* p.(723):Prentice-Hall International,Inc. ISBN-10: 0139304398.

[81] Kranofsky,G.(2005). *Some time tested tips for washing & leaching.* Chemical Engineering. Dec. Vol.112(13), p.52-56.

[82] Cussler, E.L.(2009). *Diffusion- mass transfer in fluid systems*, Cambridge university press.

[83] Seader,J.D., Henley,E.J., Roper,D.K.(2011). Separation process principles-chemical and biochemical operations. Third edition: John Wiley & Sons, Inc. ISBN 978-0-470-48183-7.

[84] *Tensile testing.* Retrieved from www.instron.us on 21-06-2012.

[85] *Tensile testing.* Retrieved from http://admet.com/test-types/tension-testing/ on 21-06-2012.

[86] Chena,Z., Moa,X., Hea,C., Wanga,H.(2008). *Intermolecular interactions in electrospun collagen–chitosan complex nanofibers.* Carbohydrate polymers, 16 May ,72(3),410–418.

[87] Hu,W., Ton-That,M.T., Perrin-Sarazin,F., and Denault,J.(2010). *An improved method for single fiber tensile test of natural fibers.* Polymer engineering and science. DOI: 10.1002/pen.

[88] Pipes,B.R., Blake,R.A,Jr., Gillespie,J.W,Jr., Carlsson,L.A.(1990). *Delaware composites design encyclopedia- volume 6*, ed: Gillespie, J.W,Jr., and Carlsson, L.A. Publishers: Technomic Publications.

[89] Zhang,Y.P., Wang,X.G., Pan,N.et al.,(2002). *Weibull analysis of the tensile behaviour of fibres with geometrical irregularities.* J Mater sci,37,1401-1406.

[90] Xia,Z.P., Yu,J.Y., Cheng,L.D., Liu,L.F., and Wang,W.M.(2009). *Study on the breaking strength of jute fibres using modified weibull distribution.* Composites : Part A ,40, 54-59.

[91] Swapan,M.A., Pickering,K.L., and Fernyhough.A.(2011). *Effects of various chemical treatments on the fibre structure and tensile properties of industrial hemp fibers.* Composites : Part A, 42,888-895.

[92] Harzallah,O., Benzina.H., and Drean,J-Y.(2009). *Physical and mechanical properties of cotton fibres : single fiber failure.* Textile research journal 201080:1093. DOI: 10-1177/0040517509352525.

10 Appendices

A Equilibrium data

g.F	g Lip/2.5 g.F	new values	Total Fibre	Total lipids(g)	mg Lip/g.F	mg Lip/g solv.
4	0.04	0.054703	3.6	0.087525269	24.3125747	0.624949462
5	0.05	0.0807	4.5	0.161399034	35.86645191	0.677201933
6	0.09	0.10194	5.4	0.244655948	45.30665696	0.71688105
7	0.16	0.119899	6.3	0.335715891	53.28823661	0.728568219
8	0.16	0.135455	7.2	0.433455807	60.20129538	0.733088387
9	0.16	0.149177	8.1	0.537035988	66.30073923	0.725928025
10	0.18	0.161451	9	0.645804653	71.75607259	0.708390693
11	0.17	0.172555	9.9	0.75924117	76.6910219	0.681517766
12	0.2	0.182692	10.8	0.876919799	81.19627764	0.646160403
13	0.18	0.192017	11.7	0.998486321	85.3407118	0.603027358
14	0.21	0.20065	12.6	1.123641002	89.17785729	0.552717996
15	0.19	0.208688	13.5	1.252127091	92.75015486	0.495745819
16	0.21	0.216207	14.4	1.383722151	96.091816606	0.432555697
17	0.2	0.223269	15.3	1.518231611	99.23082426	0.363536778

B Tensile test data

B.1 10 minutes stage 1

Diameter μm	Tensile stress (MPa)	Youngs modulus(MPa)	Strain
129	15.52	397.24	0.40
181	8.91	604.10	0.74
220	10.81	285.41	0.844
146	25.19	672.92	1.155
112	23.84	896.88	0.835
185	9.011	561.842	0.66185
180	8.809	440.49	0.702
129	18.938	825.845	0.868
163	16.489	552.370	0.734
140	13.599	723.461	0.769
140	13.645	994.4125	0.538
123	26.036	752.867	1.12
202	7.425	312.707	0.667
140	14.525	684.950	0.650
152	16.423	484.060	0.843
202	8.782	220.857	0.793
140	19.843	675.342	0.926
202	9.032	266.60	0.684
163	12.974	506.456	0.626
158	16.787	1100.725	0.530
158	8.927	688.232	0.654
163	19.389	455.962	1.041
146	13.296	403.176	0.971

B.2 10 minutes stage 3

Diameter μm	Tensile stress (MPa)	Youngs modulus(MPa)	Strain
220	10.812	285.411	0.844
146	25.192	672.92	1.155
112	23.842	896.886	0.835
185	9.011	561.842	0.661
180	8.809	440.490	0.702
142	16.469	579.393	0.546
129	18.938	825.845	0.868
163	16.489	552.370	0.734
140	13.599	723.46	0.769
163	10.282	485.70	0.576
304	7.790	325.082	0.790
332	9.488	383.291	0.545
343	6.59	408.96	0.462
180	18.43	713.21	0.760
264	9.37	308.481	0.755
354	10.412	282.23	1.079
349	5.919	252.602	0.558
202	12.535	575.27	0.811
332	6.210	302.7213	0.484
366	8.897	293.342	0.844
281	13.118	485.66	0.729
304	8.781	323.818	0.592
157	6.008	519.445	0.240
123	6.9784	1117.384	0.146

B.3 60 minutes stage 1

Diameter μm	Tensile stress (MPa)	Youngs modulus(MPa)	Strain
180	9.919	737.86	0.401
200	8.59	326.802	0.803
191	14.423	525.38	0.966
208	8.834	349.202	0.488
146	14.47	842.308	0.476
213	13.331	292.2503	1.099
265	5.988	303.334	0.603
135	25.730	649.734	0.765
247	8.59	272.669	0.610
213	14.530	371.08	0.783
304	9.622	157.587	0.958
219	12.955	312.638	0.856
146	25.737	936.92	0.928
236	10.890	473.106	0.818
236	12.968	377.934	0.733
281	4.169	269.659	0.404
191	19.110	403.551	0.739
191	17.428	511.252	0.707
197	13.785	595.93	0.754
225	12.080	674.32	0.758
225	10.960	510.98	0.536
219	11.446	495.24	0.597

B.4 60 minutes stage 3

Diameter μm	Tensile stress (MPa)	Youngs modulus(MPa)	Strain
126	37.05	858.42	0.644
143	10.260	503.611	0.564
174	9.675	377.57	0.746
233	5.817	333.031	0.6716
157	17.087	667.608	0.660
173	11.689	500.94	0.684
155	16.464	976.289	0.551
189.19	7.849	447.635	0.529
157.65	19.24	1042.49	0.606
188.18	3.567	899.91	0.5233
171.89	20.60	721.801	1.016
183.83	19.623	754.379	0.520
161.03	14.04	770.96	0.61959
152.02	12.693	461.589	0.7035
169.6	15.260	748.633	0.6567
175.54	19.697	589.764	0.9643
163.28	20.706	593.66	0.861
152.02	14.708	596.600	0.8075
180.8	17.865	563.769	0.900
164.15	20.177	699.667	0.844
146.39	15.919	813.405	0.653
174.54	9.369	589.067	0.479
202.7	16.309	651.14	0.8039

B.5 Decontaminated

Diameter μm	Tensile stress (MPa)	Youngs modulus(MPa)	Strain
152	35.29	327.23	1.019
143	33.15	251.51	0.914
128	25.80	1055.50	0.672
206	19.29	172.89	0.8518
236	15.233	163.243	0.926
312	14.339	168.917	0.7982
175	27.606	177.97	1.181
197	22.630	154.77	1.096
232	13.65	158.06	0.87
207	9.69	60.86	0.993
213	11.33	126	0.824
218	17.225	143.27	0.955
194	16.035	158.621	0.702
227	13.598	140.70	0.831
306	9.604	77.519	0.898
201	20.85	197.67	0.935
179	25.98	229.86	1.019
321	10.31	116.146	0.861
179	17.594	181.463	0.945
321	6.316	75.044	0.693
233	16.107	95.318	1.035
360	13.540	181.621	0.705
256	12.840	111.94	0.871