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# BIOFIBRE PRODUCTION FROM CHICKEN FEATHER

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

అంతు

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The University of Waikato 2011

## Abstract

The global poultry industry generates at least 2 million tonnes of chicken feather every year. Feathers are currently hydrolysed into meal used for animal feed and fertiliser. Feather consists of 91% keratin, 1% lipid and 8% water. Raw feather also contains preen oil, offal, faecal matter and poultry processing water. Its morphology consists of barbs extending at an angle from a central hollow rachis. Impurities coat the entire feather, and particulates are trapped by layers of barbules and hooked barbicels holding adjacent barbs together. These substructures present an extensive and tortuous hydrophobic surface. Feather fibre is a multipurpose, cost effective reinforcement for polymer composites. Its incorporation in plastic, wood, concrete and cardboard makes the product lighter, insulate from heat loss and improve sound attenuation.

The objective of this study was to develop a process to produce clean fibre recovered from chicken feather. In the treatment process, the heterogeneous characteristics of feather had to be considered. Raw feather was suspended in 25 L water in a pulper to be decontaminated using 2 stages of 0.1485% sodium hypochlorite adjusted to pH 10.0 with 1 M sodium hydroxide and cleaned in 3 stages of 0.15% hydrogen peroxide. The pulper disc impeller agitated the suspension at 10 Hz for 30 minute at each stage. Bacteriological tests confirmed pathogens such as *Campylobacter*, *Salmonella* and *Enterobacteriaceae* were removed during treatment. Off-white clean feathers were more than 10% whiter than dull yellow raw feather.

Cleaned feather was comminuted in 300 L water using a centrifugal pump at a flow rate of 30 Hz on full recycle for 4 hours. Rachis and partially cut feather were removed using a 5 mm aperture filter and fibre was recovered using a 1 mm filter. Wet fibre was dried to constant mass in an air-forced oven at 70°C. Fibre yield was 27% of feather input, or 54% of theoretical yield. Surface morphology showed no damage.

# Acknowledgement

I would like to express my appreciation to the following people for their support during my studies.

Firstly, I would like to thank my family for many years of care. I am grateful for the opportunities you have made possible through your sacrifices and values you have instilled in me. Thank you to friends for your sound advice, patience and being good company.

I would like to thank my academic supervisors Dr. Johan Verbeek, Assoc. Prof. Kim Pickering and Dr. Mark Lay. I am grateful for your inspirational guidance in making this project a valuable learning experience.

Thank you to Wallace Corporation, especially mentor Neville Cross and plant manager Ashley Boon, for providing industry support.

I would also like to thank Dr. Michael Walmsley, Dr. Martin Atkins, and Timothy Walmsley from the Energy Research Group for their insight into processing operations in the pulp and paper industry

Thank you for technical support from the engineering department to Stewart Finlay, Alan Smith, Yuangi Zhang, Chris Wang, Lisa Li, Indhar Singh and Luke van de Pas.

Thank you also to Wendy Jackson from the chemistry department for Soxhlet Extraction facilities, Dr. Barry O'Brien from the biology department for microscopy advice and Helen Turner for Scanning Electron Microscopy facilities.

Finally, I would like to thank TechNZ for granting me a Capability Fellowship for financial support.

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1 Introduction

# 1 Introduction

Polymers are used in almost every area of society such as packaging, transport, construction and casings. Polymers are easily shaped by extrusion, injection moulding, vacuum forming or foaming. It is durable, environmentally resistant, tough and light. Tailoring mechanical polymer properties for specific purposes often require fibre reinforcement. Common synthetic fibres include carbon, aramid and glass while natural fibres such as wood, hemp and sisal are have also been shown to be effective. An alternative to the aforementioned fibres is fibre recovered from chicken feathers, as they are widely available and has good mechanical properties [1].

New Zealand exported NZ\$6.5 billion worth of meat products, NZ\$1.2 billion coproducts and 150,000 tonnes of poultry products in 2010 [2, 3]. The major byproduct in poultry processing is chicken feather, of which Wallace Corporation (Waitoa, Morrisville) processes around 10,400 tonnes of wet chicken feather every year. Chicken feather is rendered and ground into feather meal as an additive for feed stock or as a fertiliser, it currently sells for NZ\$569 – 622 per tonne [4].

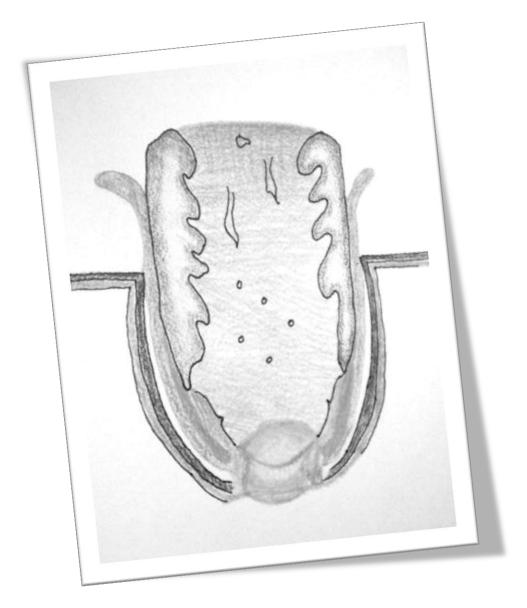
Feather structure is an ordered hierarchy comprising a central rachis from which barbs extend at an angle, with parallel barbules held together by hooked barbicels. The barbs are approximately 15 to 110 µm wide and 3 to 13 mm long, resulting in an average aspect ratio of 212. Barbules are 4 to 10 µm wide, 60 µm to 1 mm long, resulting in an average aspect ratio of 61. Barbs have an average tensile strength of 113 MPa, with an average elastic modulus of 2.8 GPa and has a 7.7% elongation at break [5].

Chicken feather is approximately 91% keratin, which is rich in cysteine bonds and its hydrophobic side chains make it waterproof. Keratin is tough, strong and chemically resistant, but as a feed additive it lacks methionine, histidine and lysine, which are essential nutrients for animals. As a fertiliser, it contains excess nitrogen [1].

Raw chicken feather contain high levels of *Salmonella*, *Campylobacter* and *Enterobacteriaceae* [6]. In addition, they are discoloured with process water, offal, chicken feed, excrement, and coated with lipids from the preen gland and

epidermal sebaceous secretions.

Useable fibres are recovered barbs removed from the rachis of decontaminated and cleaned feathers. The objective of this study was to assess various decontamination and cleaning strategies, thereby identifying suitable chemicals and concentrations thereof. Secondly, a suitable comminution process will be identified and tested for its efficiency to produce fibres suitable for use in polymer composites.



2 Chicken Feather

## 2 Chicken Feather

## 2.1 The Poultry Industry

The global poultry industry is enormous, producing approximately 2 million tonnes of chicken feather every year. There are many organisations involved in this global industry, with the World's Poultry Science Association (WPSA) representing a research network dealing with issues such as breeding, nutrition, welfare, husbandry, production, processing, product development, physiology, product quality and economics [7].

In the United States, the Department of Agriculture (USDA) is responsible for developing food information, economic opportunities and monitoring sustainable natural resource use. The US poultry industry produced 22 million tonnes broiler chickens in 2010, stimulating research into manufacturing processes for recovering fibre from chicken feather to produce specialty products [8, 9].

In New Zealand, the Institute of Food Science and Technology (NZIFST) links the food industry to researchers dealing with food processing and distribution and provides news and information about food manufacturing, packaging, processing and safety [10]. More specifically, institutions like AgResearch (formerly The Meat Industry Research Institute of New Zealand or MIRINZ) deals with ways to add value to bio-based products by increasing market access, ensuring food safety and prolonging shelf-life [11].

Poultry meat producers are represented by The Poultry Industry Association of New Zealand (PIANZ) ensure standards compliance, consumer wellbeing and animal welfare. Its main task is to maintain industry viability and to prevent avian disease outbreaks [12]. PIANZ works closely with The Ministry of Agriculture and Forestry (MAF), which manages the primary sector for farmers, food and bio-based product manufacturers and exporters. It is tasked to prevent biosecurity threats that could be damaging the environment, ensuring New Zealanders can benefit from sustainable use of natural resources [13]. Poultry products are also monitored by AssureQuality New Zealand, a commercial company providing food inspection, certification and diagnostics for the primary production sector [14].

Major poultry producers in New Zealand are Inghams Enterprises (NZ) Pty. Ltd., Tegel Foods Ltd., and PH van den Brink Ltd., which comprise 90% of poultry production in New Zealand. Inghams is a Waikato-based Australasian subsidiary employing roughly 1,000 people in New Zealand and have eleven feed mills, ten primary processing plants and nine processing plants that also make pet food ingredients [15]. Tegel Foods is a fully integrated poultry producer doing poultry breeding, hatching, processing, marketing and distribution, it is owned by the multinational Affinity Equity Partners. They have four main processing plants and employ about 1,700 people [16]. Brinks Chicken is a family business selling retail fresh and frozen chicken and specialty chicken products [17]. Kakariki Proteins, a subsidiary of Turk's Poultry Farm Ltd. (est. 1966) in Foxton, Kapiti produces eggs, poultry and does offal rendering [18]. In Waikato, NZ, Wallace Corporation is one of the largest renderers of chicken off and is the largest producer of chicken feather.

#### 2.1.1 Poultry Processing

Meat chickens are called broilers (Figure 1), while egg producing hens are layers. Breeders are selected for rapid growth rate and an efficient feed to weight gain ratio. Chicks can grow from a hatch weight of 45 g to 2.2 kg after 42 to 45 days at which point they are slaughtered and processed [19].



Figure 1: Broiler chickens in poultry farm.

Poultry processing is summarised in Figure 2, starting with live birds up to meat packaging and storage. After killing, feathers are removed by mechanical pluckers fitted with rubber fingers on rotating discs and the process is finished by operators

called pinners, who manually finish plucking. Feathers are pumped over a separation screen into a container yielding a mixture of dilute blood, grease, cleaning water and feathers.

Wastes By-Products

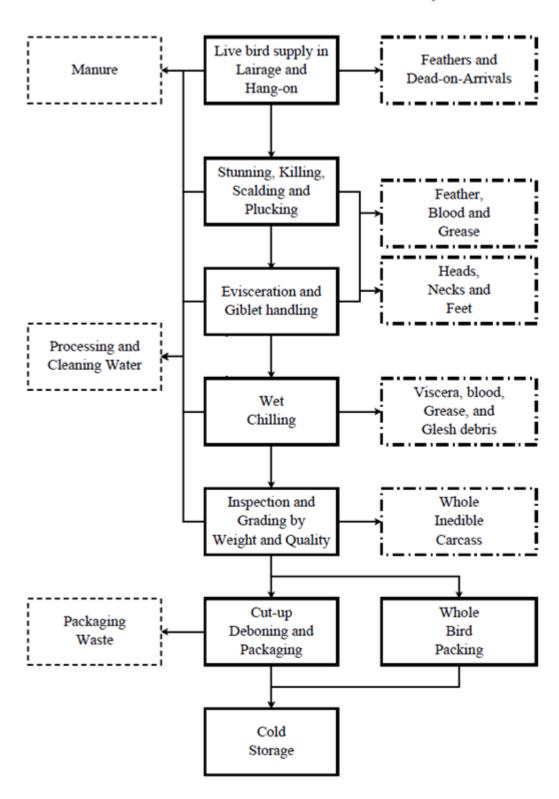


Figure 2: Typical poultry processing procedure [14].

## 2.1.2 Poultry Rendering

Parts not suitable for human consumption are shown in Figure 3A which includes the viscera (or internal organs), while Figure 3B depicts the skeletal structure. Heads, beaks and feet are often mixed up with the feather. Whole chicken and edible cuts are shown in Figure 3C.

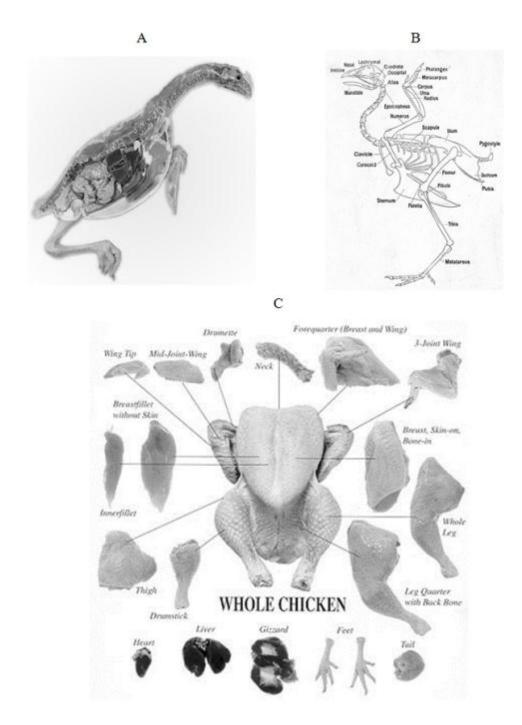


Figure 3: Poultry anatomy [20]. A – Chicken internal organs. B – Skeletal structure. C – Whole Chicken and edible cuts.

Feathers, blood, offal and trims are rendered into meal by an energy-intensive pressure cooking process at 142°C for 30 minutes. Feather meal is not a complete animal food because it is missing essential amino acids, and is indigestible unless hydrolysed. Commercial feather meal has a composition shown in Table 1. Amino acid supplements are often added to improve feed quality, resulting in animal feed comparable to soybean meal [21].

Table 1: Poultry by-product composition [3]

By-Product	Poultry blood meal	Feather meal	Poultry by-product meal
Moisture	7.0	7.0	7.0
Protein	88.9	81.0	60.0
Fat	1.0	7.0	13.0
Calcium	0.41	0.33	3.00
Phosphorus	0.30	0.55	1.70

Feather not converted to animal feed is buried or burned. Incineration releases dioxin to the atmosphere and leachate from agricultural land disposal could lead to nitrate contamination in ground water.

New Zealand renderers are exploring opportunities to increase revenue by developing alternative applications for their products. Some applications are as feedstock for plastic production, adsorbent for bioremediation, enzyme additives for detergents, antimicrobial fertiliser and a natural alternative for parts of the leather tanning process [22]. Feather, blood, fat and carcass tissues should be considered by-products with potential to increase profit rather than waste streams for disposal [23].

## 2.2 Feather Morphology

Feather makes up about 8.5% of a chicken's mass. Feathers function as insulation, protection, waterproofing, colouration and flight [24]. An epidermal sheath develops to form a short basal tube, or calamus and the main shaft or rachis. Barbs extend at an angle followed by barbules. Barbules have hooks called barbicels to connect with barbules on an adjacent barb. Muscle fibres connect the shaft bases to raise and lower feathers (Figure 4) [25].

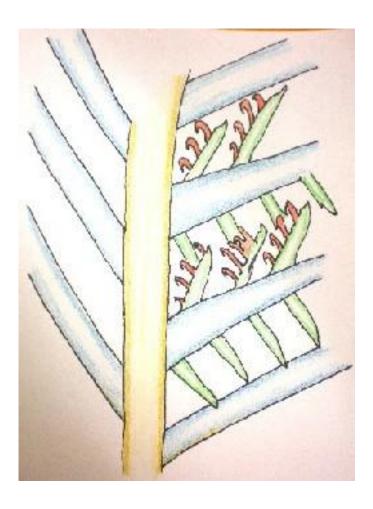


Figure 4: Feather hierarchical structure. Central shaft – rachis, Angled extensions – barbs, and barbules and barbicels [25].

## 2.2.1 Development and Differentiation

Feather, or avian integument, develops from the rachis, barbules then small hooked cilia, described in Figure 5. The follicle has a dermal papilla and an epidermal collar. The bone morphogenetic protein (BMP4) promotes rachis formation and barb fusion while the noggin enhances rachis and barb branching. The Sonic hedgehog (Shh) gene induces marginal plate epithelia apoptosis to form spaces between barbs [26].

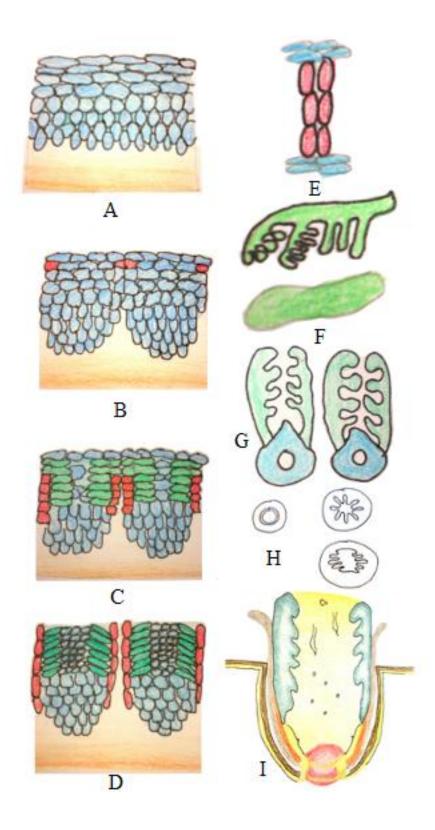


Figure 5: Feather formation [26]. A – Epithelial cells above mesenchymal cells. B – Noggin and BMP4 promote barb ridge formation. C – Shh promote barbules and rami formation. D – BMP4 and BMP2 signal for barb and barbules to separate. E – Apoptosis by Shh. F – Differentiation by BMP4 and BMP2 forms barbs and barbules or no barbs. G – Differentiated feather top view. H – Another top view showing follicles developing into barbs or rachis with barbs. I – Feather follicle cross section.

#### 2.2.2 Classification

Figure 6 illustrate types of feathers. Contour feathers (Figure 6A) cover the body surface and include flight feather which are constantly rearranged with sebum from the uropygeal, or preen gland to ensure barbules stay interlocked. Powder feathers are white particles that waterproof contour feathers. Down feathers (Figure 6B) provide insulation and do not have barbicels. Semi-plumes (Figure 6C) are similar to, but larger than down feathers. Filo-plumes (Figure 6D), at the base of every contour feather are rich in nerve endings that relate proprioceptive input or feather position [25].

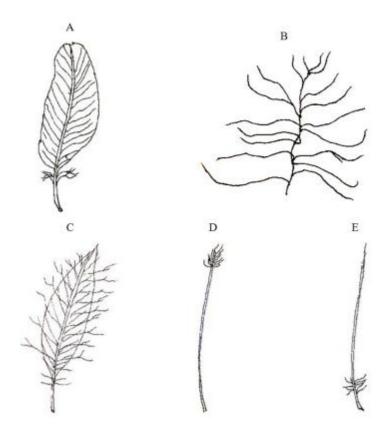


Figure 6: Feather classification. A – Contour feather maintains shape. B – Down feather provides insulation. C – Semi-plumes are a cross between contour and down. D – Filo-plumes have nerve endings at the tip to help with motion. E – Bristles around the eyes and beak acts as tactile sensors.

#### 2.2.3 Keratin

Chicken feather is approximately 91% keratin, 8% water and 1% lipids by mass [22]. Keratin is a protein with 95 amino acids and has a molecular weight of 10.168 kDa. About half the amino acids found in feather keratin are hydrophilic, but the waterproof nature of the fibre implies exterior hydrophobic side chains. Figure 7 shows the chemical structures of amino acids found in keratin. Keratin

contains about 12% serine, a polar amino acid as well as 7% of hydrophobic amino acids, valine and leucine. Aspartic acid has a negatively charged side chain, while only trace amounts of electrically charged histidine and lysine are present and no methionine at all. In Figure A 1 to Figure A 4 the amino acid composition of proteins found in different feather components are shown [27].

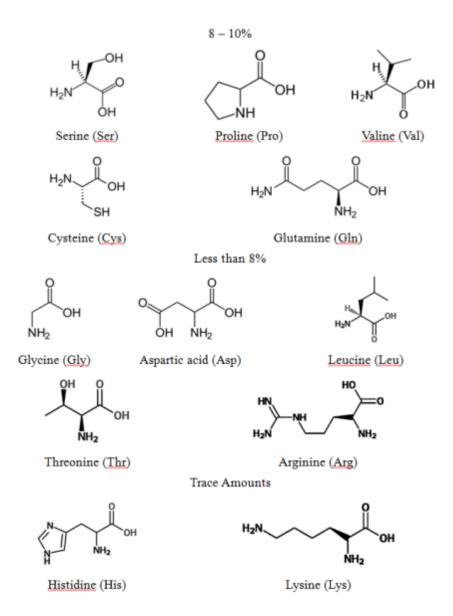


Figure 7: Amino acid chemical skeletal structures.

Infrared spectroscopy and x-ray crystallography suggest rachis to have more  $\beta$ -sheet keratins aligned in arrayed layers, while the barbs, barbules and barbicels are in ordered  $\alpha$ -helix conformation [1]. Keratin filaments are right-handed helices with four repeating units per turn. Each unit has two left-hand-twisted  $\beta$ -sheets connected by a perpendicular diad. The filament has internal hydrophobic residues

and external charged cysteine residues [28].

#### **2.2.4** Lipids

The preen gland secretes lipids to maintain the feather's physical properties such as waterproofing and colour. Pigment cells called melanocytes are responsible for the dull yellow colour of feather [29]. Free fatty acids from lipid decomposition can lead to pH changes and lead to microbial growth after slaughter. Preen oil composition is affected by age, gender, diet and contributes to feather taste. The secretion has antimicrobial properties against *Bacillus lichenformis*, which would degrade keratin. Other chemicals contributing to aroma, such as alcohols, ketones and diones attracts mosquitos [30]. Lipids extracted from different components and analysed using quantitative thin-layer chromatography are shown in Table 2 [31].

Table 2: Lipids in chicken keratin, tissue and skin [31].

Chicken Component	Lipids
Keratin	Ceramides, acylceramides, cholesteryl sulfate, glucosylsterols
	and acylglucosylsterols
Tissue	Lots: Wax diesters, triglyceride and free sterols
	Some: Phospholipids, steryl ester and free fatty acids
Full thickness	Acylglucosylceramides
epidermis	

## 2.3 Comparison with Other Fibre

Very few studies have looked into the properties of chicken feather fibres; Yang and Shah have studied fibre supplied by Featherfiber Corporation using a patented process [32]. The properties of the fibre recovered are summarised in Table 3.

Table 3: Chicken feather fibre properties [5].

Property	Va	alue
Density (g cm <sup>-1</sup> )	0.89	
Modulus (GPa)	2.8	
Strength (MPa)	113	
Elongation (%)	7.7	
Component	Barbs	Barbules
Diameter (μm)	15 - 110	4 - 10
Length (mm)	3 - 13	0.060 - 1
Aspect ratio	212	61

## 2.3.1 Synthetic Fibre

Some physical and mechanical properties of synthetic fibres are listed in Table 4.

Synthetic fibres have higher modulus and strength, implying that feather fibre by itself is not suitable for high performance applications. It is much less dense than synthetic fibres, making it an excellent candidate for light-weight composites such as car panels.

Table 4: Synthetic fibre properties [11]

Name	Density (g cm <sup>-1</sup> )	Modulus (GPa)	Strength (MPa)	Elongation (%)
Aramid	1.4	63 - 67	3000 - 3150	3.3 - 3.7
Carbon	1.4	230 - 240	4000	1.4 - 1.8
E-glass	2.5	70	2000 - 3500	2.5
Nylon 6	1.09 - 1.14	200 - 300	35 - 70	20 - 50
Saran	1.67 - 1.71	100 - 150	30 - 40	20 - 40
S-glass	2.5	86	4570	2.8
Vinal	1.25 - 1.35	300 - 1200	50 - 75	17 - 26
Vinyon	1.34 - 1.36	350 - 450	25 - 30	20 - 30

#### 2.3.2 Plant Fibre

More than 29 million tonnes of plant fibre is produced worldwide each year. A selection of plant fibre and estimated annual production are listed in Table 5. Some of these plant fibres are the main source of income for farmers in developing countries. Fibres like abaca, sisal and coir earns \$30, \$75 and \$100 million per year, respectively [33].

Table 5: Plant fibre species, annual production and earning [33].

Name	Species	Annual Production (MT)
Plant Fibre		Approximately 29
Abaca	Musa textiles	0.07
Coir	Cocos nucifera	0.5
Cotton	Gossypium genus	25
Flax	Linum usitatissimum	0.147
Hemp	Cannabis sativa	0.113
Jute	Corchorus capsularis	2.3 to 2.8
Ramie	Boehmeriz nivea	0.28
Sisal	Agave sisalana	0.3

Table 6 shows feather barbs have similar diameter range to most plant fibres, while barbules are much thinner. Plant fibres can be several metres long, but feather barb lengths are comparable to short cotton fibres, which is the most commonly used fibre for fabric.

Table 6: Plant fibre physical properties [4].

Name	Diameter (µm) [33]	Length (m) [33]	Density (g cm <sup>-1</sup> )
<b>Plant Fibre</b>	11 - 300		1.15 – 1.55
Abaca [11]	17 - 21	Up to 3	-
Coir	17 - 21	Up to 0.35	1.15 - 1.46
Cotton	11 - 22	0.01 to 0.065	1.5
Flax [10]	17 - 21	0.025	1.5
Hemp [34]	18 - 38	18 - 40	1.5
Jute	16 - 20	1 to 4	1.46
Ramie [35]	25 - 30	0.012	1.55
Sisal	100 - 300	Up to 1	1.45

The mechanical properties of some plant fibres are listed in Table 7. Feather fibre has lower modulus, or stiffness, and similar tensile strength as coir. Plant fibres have at least 2 times higher tensile strength.

Table 7: Plant fibre mechanical properties [4]

Name	Modulus (GPa)	Strength (MPa)	Elongation (%)
Plant Fibre	4 - 128	130 - 1500	1.2 - 40
Abaca [11]	33.6	813	-
Coir	4 - 6	130 - 220	15 - 40
Cotton	5.5 - 27.6	300 - 1500	3 - 8
Flax	27.6	345 - 1500	2.7 - 3.2
Hemp [34]	70	690	1.6
Jute	10 - 55	200 - 800	1.16 - 8
Ramie [35]	61.4 - 128	400 - 938	1.2 - 3.8
Sisal	9.4 - 22	468 - 700	2 - 7

#### 2.3.3 Animal Fibre

About 2.3 million tonnes of animal fibre is produced in the world every year, most of which is wool from sheep. Alpaca wool has a market share of \$50 million a year, while wool earns about \$80 billion a year. Annual production of some animal-based fibres is listed in Table 8.

Table 8: Animal fibre species, annual production and earning [33]

Name	Species	Annual Production (kT)
Animal Fibre		2,300
Alpaca wool	Lama pacos	6.5
Angora wool	Oryctolagus cuniculus	2.5 - 3.0
Camel hair	Camelus bactrianus	2
Cashmere	Capra hircus laniger	15 - 20
Mohair	Capra hircus	5
Silk	Bombyx mori	150
Wool	Ovis aries	2,100

The material properties of some animal fibre are listed in Table 9. The moisture regain of silk and wool are 11% and 13.6%, respectively. Feather fibre is

relatively finer than camel hair and wool.

Table 9: Animal fibre physical properties [4]

Name	Diameter (μm)	Length	Density (g cm <sup>-1</sup> )
Camel hair	20 - 105	8.89 cm	1.32
Silk	10 - 13	1 - 1.5  km	0.93
Wool	25.5 - 28.5	8.255 cm	1.31
Feather barb	15 - 110	3 - 13  mm	0.89

The mechanical properties of a few animal fibres are listed in Table 10. Feather barb fibre has stiffness comparable to upper limits of camel and wool and slightly lower strength than other animal fibres.

Table 10: Animal fibre mechanical properties

Name	Modulus (GPa)	Strength (MPa)	Elongation (%)
Camel hair	2.02 - 3.87	121 – 212	37 – 50
Silk	0.015 - 0.017	610 - 690	4 - 16
Wool	2.3 - 3.4	120 - 174	25 - 35
Feather	2.8	113	7.7

These properties indicated that feather fibre is a superior lightweight filler for textile products such as pillows, duvets and upholstery.

## 2.4 Feather Fibre Applications

Feather fibre finds wide spread application. Traditionally, feathers have been used as thermal insulation in products such as jackets or as volumetric filler in pillows. More recently, it has been recognised that more valuable applications are possible, such as biodegradable plastics and filter media.

From the late 1970's patent literature would suggest that poultry feathers were dry-cleaned and used for quilts, pillow filling and jacket insulations [36, 37]. Fibre production from chicken feather has not been commercialised in New Zealand, and current practice is to hydrolyse feather into feather meal for animal feed and dispose surplus feather by composting. Some recent applications of feathers are further discussed below.

#### 2.4.1 Polymer and Composites

Insulation made from ground chicken quill and polypropylene (PP) showed good sound attenuation properties. The noise reduction coefficient (NRC) from ground

quill composites was 71% greater than jute composite. Factors such as inner and outer void content of the quill contributed to better sound absorption, making quills a promising noise insulation panel and head-liner substrate [38].

Building material containing chicken feather fibre, recycled kraft paper and newspaper was made into non-woven mats by a wet-lay process. The fibre mats were infiltrated with acrylated epoxidised soybean oil (AESO) resin using vacuum assisted resin transfer moulding (VARTM). The product is cheap, environmentally friendly, and energy efficient. Product applications include temporary building structure and light vehicle bodies [39].

Recent work by the US Agricultural Research Services produced dashboards, soundproof materials, plant pots and 5 µm mesh depth filters. The patented and licensed technology has a pilot plant production capacity of 90 kg fibre per hour. The planned large-scale facility is set to produce 5 tonnes per hour and will be located in Southwest Missouri or East Maryland [7].

Printed circuit boards for the electronics industry can be made from keratin feather fibres infused with acrylated epoxidised soybean oil (AESO). In the study it was shown that keratin fibres remained hollow resulting in a dielectric constant (k) from 1.7 – 2.7. This value is lower than silicon dioxide, epoxy and polymer dielectric insulators [40]. The chicken feather fibre-epoxy composite was compared with E-glass-epoxy composites. Boards containing chicken feather fibre had a 2 to 4 magnitude greater electrical resistance than their E-glass counterpart. The dielectric constant values were comparable to commercial printed circuit boards [41].

It has been shown that potassium hydroxide hydrolyses feather, making it suitable for applications such as biopolymer films, foils and drug encapsulation materials [24]. Furthermore, bacteria such as *Streptomyces* and *Bacillus* sp. can hydrolyse keratin to enhance drug delivery as biocompatible materials typically inserted into animal tissue. It also has a novel potential to hydrolyse prions, misfolded proteins that cause bovine spongiform encephalopathy and Creutzfeldt–Jakob disease in humans [42].

#### 2.4.2 Adsorbent in Bioremediation

Chicken feather has exceptional adsorption properties. Chicken feather has been utilised as low-cost adsorbents on a fixed-bed system for phenol removal from aqueous solutions [43]. Filter media made from chicken feather accumulated gold-cyanide ion from aqueous solutions with a high yield [44]. Chicken feather also complexes with palladium (Pd), a precious metal [45]. Regenerated chicken feather adsorbed hexavalent chromium (Cr<sup>6+</sup>) ions in wastewater with high efficiency [46]. Arsenite (AsO<sub>4</sub><sup>3-</sup>) can be removed from aqueous solution by direct adsorption onto chicken feather, while traditional bioremediation required an oxidation step to form arsenic first. The sulfhydryl groups on cystein/cysteine molecules forms a complex to take up As<sup>3+</sup> [47, 48].

Keratin pyrolysis produced porous carbon-nitrogen fibres for nanotechnology. The heat-resistant adsorbent product had accessible pores with a diameter of less than 1 nm and can also be used for hydrogen storage or small gas molecule separation. The micro-pores sizes were more uniform than commercial activated carbon [49, 50].

## 2.4.3 Enzymatic Applications

Chicken feather substrate can be used to produce enzymes other than keratinase. Azocasein, a dye used to induce inflammation in animal testing was degraded by *Bacillus licheniformis* ER-15 cloned into *E. coli* HB101 via vector pEZZ18 ker BL2 [51]. *B. halodurans* JB 99[52], *B. pumilus* A1 [53, 54] and *B. licheniformis* RPk [55] secreted caseinase, used in the human body to digest milk. *B. licheniformis* NH1 secreted α-amylase to convert starch to sugar [56].

Feather used as a substrate in microbial cultures that secrete extracellular enzymes for detergent formulations. Serine protease from *Bacillus mojavensis* A21 is compatible with a variety of alkaline surfactants at 60°C. The enzyme can be used with non-ionic and anionic surfactants as both solid and liquid detergent. The formulations remain active and stable at warm temperatures [57].

#### 2.4.4 Crop Fertiliser and Plant Antimicrobial Control

Bacteria feeding on as little as 0.1% w/v feather is able to promote plant growth such as siderophore (iron transport), indoleacetic acid (phytohormone for cell

division), ammonification, hydrolytic enzyme activity and fungal growth inhibition. Examples include *Xanthomonas sp.* P5 [58] and *Stenotrophomonas maltophilia* from rhizospheric soil of reed growing on chicken feather [59]. *Bacillus subtilis* from forest soil also exhibited phosphate solubilisation and broad-spectrum antimicrobial activity [60].

#### 2.4.5 Animal Products

Synthetic tanning methods use toxic chemicals, but the naturally occurring enzymes from *Bacillus halodurans* JB 99 can remove hair from animal during leather manufacture [52]. Another enzyme, secreted by *B. halodurans* PPKS-2, was isolated from rice mill effluent and immobilised. Its activity was not inhibited by SDS, EDTA or H<sub>2</sub>O<sub>2</sub>. The immobilised enzyme was used on goat skin and collagen integrity remained after treatment [61]. *Bacillus pumilus* CBS degraded 80% of goat hair and 68% of bovine hair after 24 h at 37°C [62]. In a related process, chicken feather was transamidified, or had its peptides exchanged with amines. The polypeptides were acylated with maleic anhydride and copolymerised with acrylic acid. The product was applied to goat skin wet-blues, hides that have been processed with chromium tanning agents. The crust, or leather before colouring, had improved strength and were not damaged by dyes [63]. *Aspergillus oryzae* secreted keratinase that can be immobilised on sintered glass beads. The product acted on breaking down bovine serum albumin, casein, keratin, chicken feathers, collagen, duck feather and sheep wool [64].

Candida parapsilosis feeding on feather secretes a serine peptidase that hydrolyses collagen into gelatine [65]. Bacillus megaterium F7-1 feeding on autoclaved chicken feather produced keratinase that readily degraded feather, meat, duck feather and human nail, but not human hair and sheep wool [66]. These enzymes are being used in wool wash in the laundry detergent industry and keratin hair treatments.

## 2.5 Fibre Production from Feather

Recovering fibre from chicken feather that are usable for the aforementioned applications require some degree of processing. Most importantly, feathers require decontamination to remove pathogens, cleaning to remove residues from chicken

processing and in some cases size reduction; these are discussed in more detail in the following sections.

#### 2.5.1 Decontamination

Unprocessed feather requires pre-treatment, starting with decontamination to ensure process hygiene and cleaning to remove impurities that cause objectionable odour, discolouration and equipment fouling.

Chickens are warm-blooded, leading to a variety of microorganisms on chicken parts, including mesophilic or psychrotrophic organisms that originate from the animal, its habitat, processing equipment and human handlers. Contamination with pathogenic bacteria described in Table A 4 may lead to spoilage and gastroenteritis; a carcass surface could contain as much as 100 to 1,000 mesophilic bacteria per cm<sup>2</sup> [6].

Two common *Campylobacter* species are *C. jejuni* and *C. coli*, where growth occurs between 25 to 43°C. Gastroenteritis could result from contact with as few as 500 cells of *C. jejuni* [67]. They are found on most animals, especially poultry, but the hosts do not show symptoms [68]. The *Enterobacteriaceae* family is found in the intestines of chickens, but handling during slaughter causes crosscontamination. Common genera include *Enterobacter*, *Escherichia* and *Salmonella*. The psychrotrophic *Salmonella* persist even when the meat is chilled or frozen [69]. *Enterobacteriaceae* detection is used to indicate food quality and safety [70].

Chlorine bleach such as 1 - 5% w/v sodium hypochlorite (NaOCl) at pH 10.0 to 12.0 at  $25^{\circ}$ C can make unprocessed feather bacteriostatic, where colony proliferation is halted or delayed [71].

## 2.5.2 Cleaning

Feather is traditionally washed by organic liquids such as chlorinated hydrocarbons, and has been described in patented literature. The chemicals are reused after distillation and solid removal. C<sub>2</sub> hydrogen derivative mixtures are used such as trichloroethylene, perchloroethylene, tetrachloroethylene, and tetrachloroethene. These chemicals are used for dry-cleaning but discharge has

detrimental effects on the environment [36].

The commercialised process filed by the United States Department of Agriculture in 1998 washed feathers in polar organic solvents such as ethanol. This process required dual stage leaching, and solvent recovery required additional chemicals to break the ethanol-water azeotrope [32].

Another patent has recommended using an inorganic solvent such as hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), chlorine bleach (such as sodium hypochlorite) or detergents such as sodium dodecyl sulfate (SDS) [72]. Hydrogen peroxide is routinely used in waste water treatment, where proper use allows it to break down into innocuous water and oxygen [12].

Feather cleaning methods are often adapted from unit operations in the pulp and paper, waste treatment or food industry. Pulp washing separates dissolved organics and spent chemicals from wood fibres. Dissolved contaminants are removed from the suspension for further treatment. Well-designed pulp washing minimise effluent discharge, chemical use and spillage. The cellulose fibre suspension is mixed, filtered and fed to another washing steps in series [73]. Pulp is also bleached to alter the fibre's optical properties by removing chromatophores, or light absorbing components. Bleaching also adjusts the degree of cellulose polymerisation, but it is important that when brightness is increased that material strength is preserved [74].

In the food industry, lipids are routinely separated from plant material. Products include vegetable oils, sugars, carbohydrates, aromatics, antioxidants and cholesterol removal, which is similar to lipid removal from chicken feather. Solid-liquid extraction (leaching) is commonly used, where a solute transports fluid from a solid matrix to a solvent. It is a multi-component, multiphase, un-steady state mass transfer operation [75]. The most important steps in leaching include:

- entrance of the solvent into the solid matrix,
- solubilisation and/or breakdown of components,
- transport of the solute to the exterior of the solid matrix,
- migration of the solute to the exterior of the solid matrix,

- movement of the extract with respect to the solid (i.e. extract displacement), and
- separation and discharge of the extract and solid.

Leaching is classified into percolation or immersion methods; batch or continuous modes; and whether the process is single stage or uses multiple counter-current stages [76]. A suitable solvent for feather leaching must meet the following criteria [77]:

- compounds to be removed must be soluble in the solvent,
- the product must not contain solvent residues,
- the chemical should be recovered by distillation or evaporation without azeotrope formation and has a small latent heat of vapourisation,
- interfacial tension, viscosity and wettability should allow solvent to flow and penetrate pores and capillaries in the feather, and
- ideally, the solvent should be non-toxic, stable, non-reactive, non-flammable, harmless to the environment and cheap.

Chicken feather has an intricate heterogeneous microstructure. The rachis forms capillaries and hooked barbicels that add to resistance to lipid diffusion. Leaching is more effective when feather components have full contact with the solvent, which would otherwise lead to lipid entrainment. The solids should be agitated and reduced to uniform size by comminution to ensure a high surface area required for efficient extraction. It has also been shown that temperature control during leaching maintained feather cell membrane permeability; heating reduced lipid viscosity and ensured micro-organisms remaining inactive; however, the temperature should be low enough to prevent keratin degradation [78].

#### 2.5.3 Effluent

The waste purged from the fibre production process should be free from pathogens. Feather treatment effluent should meet regional by-laws summarised in Table A 3. Conditional trade waste consent is required for volumes greater than 5 m<sup>3</sup> per day, instantaneous flow rates greater than 2 L s<sup>-1</sup> or discharge temperature above 40°C and the waste must not interfere with sewage flow. As an example, a solvent layer settled or floating in the discharge cannot be allowed because the sewer could be subjected to corrosion or harmful gas emissions.

Furthermore, wastewater treatment can be impaired if the ratio between Chemical Oxygen Demand (COD) and five-day Biochemical Oxygen Demand (BOD<sub>5</sub>) fall beyond 1:1 to 100:1 [79, 80].

#### 2.5.4 Comminution

Size reduction, or comminution, is the process of applying mechanical stress to a solid to reduce particle size. Comminuted feather has a lower mean particle size and a narrower particle size range implying a more uniform product. Comminution is an energy intensive preparation step and up to 99% of the energy input is converted into heat and absorbed by carrier medium, product and equipment. Suitable methods include crushing, impact, cutting and using shear induced from the surrounding medium. Factors affecting comminution equipment selection include stressing mechanism, feed and product size, material properties, carrier medium, operation mode and unit operation integration [81]. Some challenges during comminution are that a heterogeneous feather mixture could result in reduced average fibre length and changes in fibre length distribution. Also, lipids remaining after cleaning could cause product discolouration.

Feather comminution could broadly be divided into dry and wet processes. Dry comminution using a comb/brush separator has been described in a patent. Fibres were combed by rotating brushes and air pulled through a screen. The screen aperture size prevented rachis from passing through [32]. One complication was that dry barbs had a static charge and stuck to dry equipment surfaces.

Wet comminution would overcome these problems. A patent recommended wet comminution by using refiners, pulpers or disc mills. A high shear pulper used a coupled rotor and stator with a close tolerance to remove fibre from rachis. The refiners, or disc mill, grinded, sheared, shredded, pulverised, rubbed and fluffed the feather into a suitable product [72].

#### 2.5.5 Filtration

After comminution fibre can be recovered using three steps:

- feather fraction less dense than water float to the top and is skimmed off,
- feather in suspension is separated by gravity filter, and

wet feather is dried.

Fibre separation classifies particles by size in a continuous process and separation is affected by particle density and shape. These properties need to be constant for effective separation and reproducible results. Other factors that need to be considered during filtration are [82]:

- particle shapes and size,
- volume and mass fraction,
- filter performance,
- whether the process is by cake and/or depth filtration,
- number of stages for filtration in series and number of filter cycles for circuits,
- the type of separation equipment such as vacuum filter, batch pressure filter, continuous pressure filter and centrifugal filters, and
- filter media selection.

A Beloit pressure screen in Figure 8, removes debris from slurry. Several units are set up in series or parallel to form a multi-stage cascade. The pressure screen shown in consists of a rotor, screen cylinder, pipes and a chamber. The rotor increases consistency, or solids concentration. The screen cylinder, or basket, can be slotted, perforated or contain wedge wires. Perforations and wedge wires allow 100% screen plate effective area. Slots usually have 60 – 65% effective area or can be up to 75% or 80%. An optimised pressure screen process improves screening efficiency, increases capacity and minimises equipment downtime due to blockage [83].

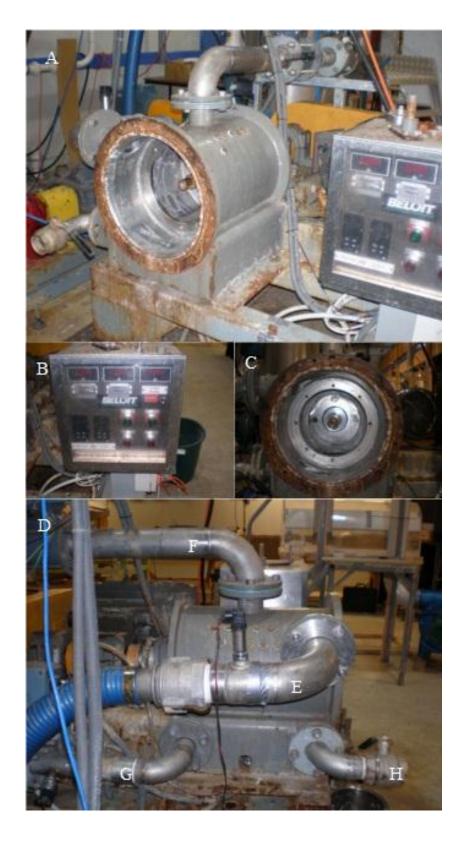
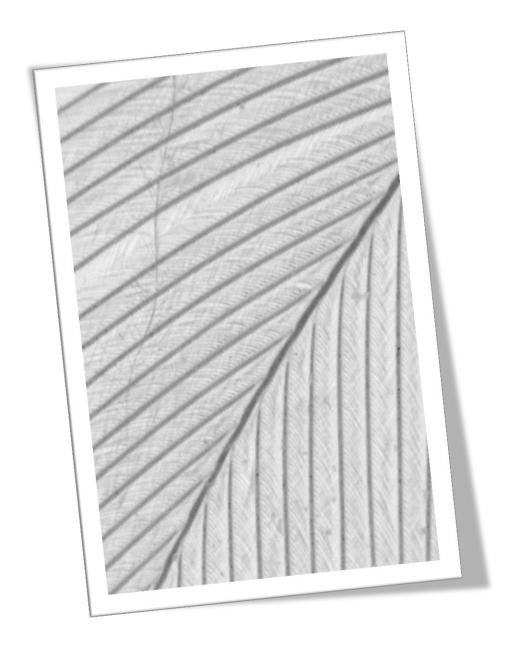


Figure 8: Beloit pressure screen. A - The open pressure screen chamber. B - Control panel. C - Front view of pressure screen chamber with basket. D - Side view. E - Inlet. F, G and H - Outlet.

## **2.5.6** Drying

Pulp fibre is dried by heated air circulation and wet fibres are often fluffed before drying to disperse particles. The dispersed fibre has greater specific surface and contact with drying medium, which facilitates efficient evaporation. Fibre dispersion also allows transport through a continuous drying system.

Wet fibre surfaces are covered with moisture at the start. The drying rate during this period is high and the fibres warm to wet-bulb temperature of the air. Internal moisture evaporate when the surface dry out. Resistance to drying increases during this phase and surface temperature rises. The drying rate depends on how liquid migrates to the surface for the specific material. The exhaust air is reheated and recycled to reduce fresh air intake. Dry fibre is cooled to prevent brightness reversion [84].



3 Experimental

# 3 Experimental

### 3.1 Materials

Chicken feather used in this project was obtained from Wallace Corporation's Waitoa rendering plant. The rendering department operates 5 days a week for 16 hours a day, processing about 200 tonnes of wet feather per week, most of which come from a close by Ingham processing plant (Figure 9). Feathers are rendered into feather meal, yielding 50 tonnes of feather meal per week. Feather generation increases by about 3% every year.



Figure 9: Raw feather receiving at Wallace Corporation's Waitoa rendering plant.

Chemicals used for decontamination were sodium hypochlorite (NaOCl) and sodium hydroxide (NaOH). For the purpose of initial scoping trials, commercial bleach with trade name Janola was used and contains 42 g L<sup>-1</sup> NaOCl (equivalent to 4% w/v available chlorine at the time of manufacture), and 4 g L<sup>-1</sup> sodium hydroxide (NaOH).

Chemicals used for feather treatment were 15% sodium hypochlorite, ethanol absolute, 30% hydrogen peroxide, Tween-80, and *n*-hexane supplied by Univar. Sodium hydroxide pellets, sodium dodecyl sulphate powder and peptone powder were supplied by Sigma Aldrich.

# 3.2 Equipment

Equipment used in this study is presented in three parts, equipment used for lab scale experiments, large scale equipment and analytical equipment.

### 3.2.1 Lab-scale

A Boltac 6-unit mixer was used for initial scoping trials for feather decontamination and cleaning (Figure 10). Each unit had a working volume of 1 L and stirring speed up to 100 rpm without temperature control.



Figure 10: Boltac 6-unit mixer (left).

A Breville BFP300 and a Kenwood FP920 blender processor (Figure 11) was used for lab scale comminution.





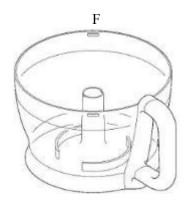


Figure 11: Lab-scale comminution equipment. A – Breville processor blade. B – Breville 3 L processor and 2 L blender. C – Kenwood 2 L blender jug and blade bowl. D – Kenwood 3 L blender bowl on rotor unit. E – Kenwood dry mill. F – Kenwood processor bowl.

## 3.2.2 Large Scale

Feather was decontaminated and cleaned using a 30 L Lamort Pulper (Figure 12A). The equipment specification is summarized in Table A 11. The number and spacing of rotor teeth can be varied for fine or coarse fibre removal and angled baffles deflect solids coming into contact with the side of the tank [1]. In this study, a flat disc agitator was used to prevent feather entanglement around the agitator (Figure 12B).

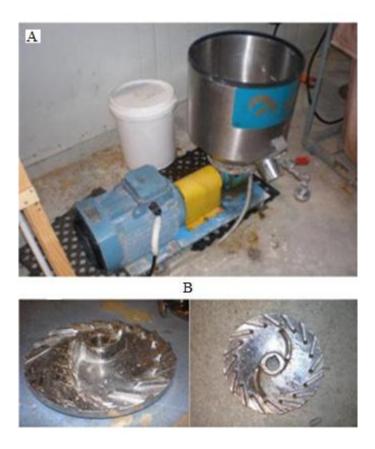


Figure 12: Lamort pulper. A – Motor and tank and B – Disc impeller.

Feather comminution was performed using a 600 L stainless steel tank combined with a centrifugal pump, operated under total recycle (Figure 13). The tank had an inner diameter of 0.7 m and an inner height of 1.8 m. The outlet of the tank was directly mounted to the suction end of the pump with a diameter of 100 mm. The discharge side was connected to a 101.6 mm diameter, 8 m long PVC pipe and was returned directly back to the tank.

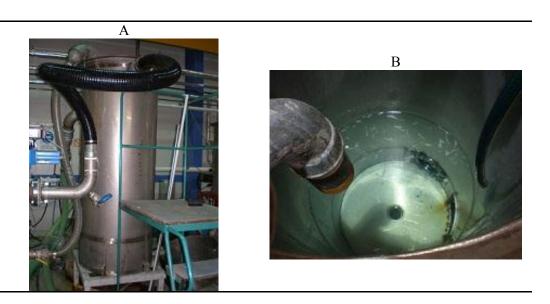




Figure 13: Comminution system. A – 600 L stainless steel tank. B – Tank filled with 300 L water. C – Scanpump connected to side of tank. C – Side access panel and discharge valve.

A centrifugal pump (Scanpump 310-0133) was to circulate the feather suspension, capable of delivering 250 m<sup>3</sup> h<sup>-1</sup> (Figure 14). Technical data are shown in Table A 12. The pump was controlled by variable speed drive (Altivar-61). The volumetric flow rate was monitored using a Promag 35 electromagnetic flow meter from Endress+Hauser.

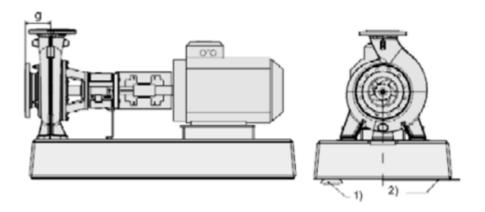


Figure 14: Scanpump 310-0133.

Chicken feather suspensions were filtered using filters shown in Figure 15 to separate fibre from partially cut and uncut feathers. Only the fine hand-held filter is shown in Figure 15 as both had a similar appearance.



Figure 15: Tall, wide and fine filters used during fibre recovery.

Details of filters used are shown in Table 11.

Table 11: Filter dimensions

Filter Type	Tall	Wide	Coarse	Fine
Diameter (m)	0.3	1.0	0.20	0.15
Container Height (m)	1.0	0.5	0.30	0.05
Aperture location	Side	Base	Base	Base
Aperture diameter (m)	0.0254	0.001	0.05	0.001

#### 3.3 Methods

## 3.3.1 Lab-scale Decontamination and Cleaning

10 g unprocessed chicken feather was agitated using the Boltac mixer at 60 rpm in 500 mL diluted Janola, ethanol, hydrogen peroxide  $(H_2O_2)$ , or sodium dodecyl sulphate (SDS) for 1 hour. Liquid were filtered away with 1 mm mesh hand held filter. The wet feathers were dried to constant mass in Contherm air-forced dryer at  $70^{\circ}$ C.

#### 3.3.2 Lab-scale Comminution

10 g chicken feather suspended in 500 mL water was cut with a Kenwood FP920 (Figure 11) horizontal double knife unit in the processor bowl, alternating angled 4-blade unit in the blender jug, and a dry mill with blades similar to the blender.

#### 3.3.3 Large Scale Decontamination

The Lamort Pulper was filled with 25 L water and 2.5 kg unprocessed feather. 1M NaOH was added to keep suspension at pH 10.0 before 250 mL 15% NaOCl was added. The suspension was agitated at 10 Hz for 30 minutes. The liquid phase was drained, replaced with 250 mL 15% NaOCl adjusted to pH 10.0, and mixed for another 30 minutes. The liquid phase was drained and the decontaminated feather was rinsed with about 25 L water to remove remaining NaOCl. The process is summarised in Figure 16.

### 3.3.4 Large Scale Cleaning

The cleaning step was a three stage process. The decontaminated feather in the Lamort Pulper was suspended with 25 L water and 125 mL 30% H<sub>2</sub>O<sub>2</sub> was added. The suspension was agitated at 10 Hz for 30 minutes. The liquid phase was drained and replaced with 25 L water and 125 mL 30% H<sub>2</sub>O<sub>2</sub> before agitation at 10 Hz for another 30 minutes for stages two and three. The clean feather was rinsed with 25 L water to remove remaining H<sub>2</sub>O<sub>2</sub>. The process is summarised in Figure 17.

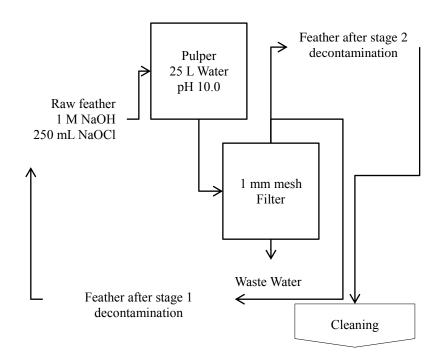


Figure 16: Decontamination block flow diagram.

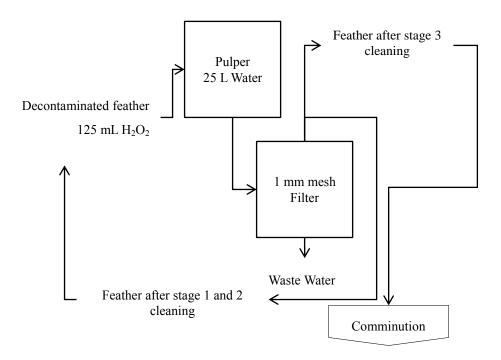


Figure 17: Cleaning block flow diagram.

### 3.3.5 Large Scale Comminution

The 600 L tank was filled with 300 L water and 1.33 kg clean feather. The centrifugal pump was set to 30 Hz impeller speed. The feather-water slurry circulated through the system for four hours. 4 L samples were taken every hour, weighed and filtered into feather fraction (> 5 mm mesh) and fibre fraction (> 1

mm mesh) and liquid phase (< 1 mm mesh). The feather and fibre were dried to constant mass in Contherm air-forced dryer at 70°C. The slurry temperature was recorded at every sample interval. The process is summarised in Figure 18.

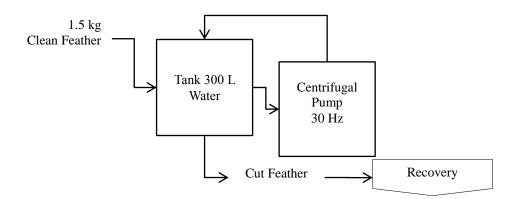


Figure 18: Comminution block flow diagram.

## 3.3.6 Large Scale Fibre Recovery

Fibre was recovered from 5 mm and 1 mm filters set up like Figure 19. The slurry was allowed to float to the surface while bulk slurry cooled to a temperature safe to handle. The floating fraction was skimmed off with a net. The suspension was discharged over the 1 mm mesh wide filter from a horizontal valve at the side of the tank near the base. Sediments were discharged through a vertical valve at the tank base and filtered by a 1 mm mesh filter. Floating feather and suspension were divided into manageable portions and washed over a 5 mm filter placed above a 1 mm filter with high pressure water.

The feather fraction remained on the 5 mm filter and the fibre fraction remained on the 1 mm filter. Feathers and fibres were laid flat on plastic trays lined with aluminium foil and dried to constant mass at 70°C in a Contherm air-forced dryer. Dry feather and fibre were weighed and sealed in plastic bags.

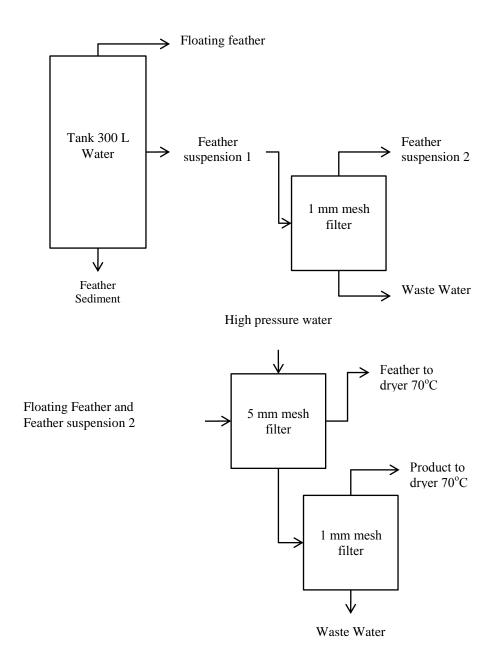


Figure 19: Fibre recovery block flow diagram.

#### 3.3.7 Analysis

Feather samples were sent to NZ Lab to test for *Campylobacter*, *Enterobacteriaceae* and *Salmonellae*. Each specimen was weighed, rinsed and removed from 400 mL of 0.1% peptone water according to APHA (4) 2.527. The bacteriological test procedures are summarised in Table A 5.

Feather morphology was observed using a Wild-Leica M2B stereo microscope with 6,4x, 16x and 40x click magnification mounted on adjustable stand and illuminated by halogen light [85]. Fibres were photographed using an Olympus PM-10M digital camera. Technical specifications for the microscope are listed in Table A 6.

A Hitachi S4100 Field Emission Scanning Electron Microscope (FE-SEM), similar to Figure 20, was used to examine feather surface properties. The specimens were coated with platinum and examined at 20 kV accelerating voltage. Micrograph magnifications used were 1,000x and 3,500x [86].

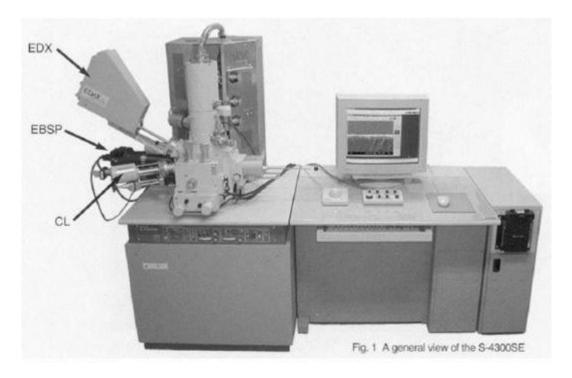


Figure 20: Hitachi S4000 series FE-SEM [3].

Soxhlet extraction was used to extract soluble impurities from chicken feather (Figure 21). The main apparatus consisted of the Soxhlet extractor, a glass chamber with siphon, a water cooled condenser and a 125 mL reservoir flask The Soxhlet extractor was connected to the reservoir flask with a condenser and

clamped above a heating element (Thermo Scientific).

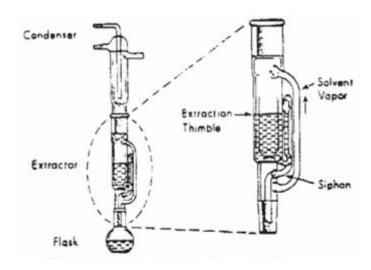


Figure 21: Soxhlet extractor apparatus [4].

Solid specimens were dried to constant mass in a Contherm air-forced oven after extraction and solvents were recovered using vacuum evaporation in a Buchi Rotavapor consisting of a rotating angled flask on a water bath, coil condenser, pressure release tap and a clamped round flask. The tests were carried out in triplicate.

Material colour was determined by Konica Minolta CR-410 Chroma Meter (Figure 22) with technical specifications in Table A 7. The pulsed xenon arc lamp (a) light source with baffle (b) and diffuser (c) allow light to travel only in the vertical direction to illuminate sample (f). The optic fibre cables (d) and (e) monitors colour and illuminant, respectively (Figure 22) [87]. The meter was calibrated with a white plate. Absolute colour readings were recorded in L\*a\*b\* space and converted to RGB using method described in Table A 8.

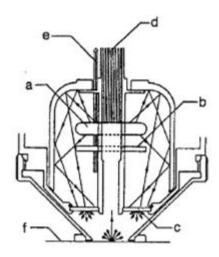


Figure 22: Konica Minolta CR410 Chroma meter sensor [6].

## 3.4 Experimental Plan

#### 3.4.1 Lab-Scale Decontamination

Information on the Janola container suggested household cleaning in 1:4, 1:8 or 1:20 dilutions for 5 to 10 minutes. 10 g unprocessed feather was treated in 500 mL diluted Janola in the concentrations listed in Table 12 for 5 or 10 min.

Table 12: Janola concentrations and residence time

Sample	Diluted Ratio	Concentration (% w/v NaOCl)	Time (min)
J1	1 in 20	0.21	5
J2	1 in 8	0.525	5
J3	1 in 4	1.25	5
J4	1 in 20	0.21	10
J5	1 in 8	0.525	10
<b>J6</b>	1 in 4	1.25	10

## 3.4.2 Lab-Scale Cleaning

10 g unprocessed chicken feather were mixed with 500 mL cleaning agents for 1 h. Initial trials with ethanol dilutions listed into Table 13 were carried out to gauge the mixing effect of Breville blender-processor.

Table 13: Low concentration ethanol cleaning

Concentration (% v/v)	Volume* (mL)	Mass (g)	Concentration (wt%)				
0	0	0					
1	5	3.95	0.79				
3	15	11.85	2.39				
5	25	19.75	3.99				
7	35	27.65	5.61				
10	50	39.50	8.07				
*ethanol pipetted into volumetric flask and filled up to 500 mL with water							

The surfactant Tween-80 (Table 14), recommended by patent [32], was also used in the blender to gauge mixing effects.

Table 14: Tween-80 cleaning

Tween-80 Volume (mL)	Concentration (% v/v)
2.5	0.005
3.5	0.007

30% hydrogen peroxide was diluted to concentrations in Table 15 recommended by patent [72].

Table 15: Hydrogen peroxide cleaning

Volume (mL)	mass (g)	Concentration (wt%)
2.75	3.0525	0.6101
2.25	2.4975	0.4993
1.75	1.9425	0.3884
1.25	1.3875	0.2774
0.75	0.8325	0.1665

Sodium dodecyl sulfate, recommended by patent [88], was diluted to concentrations listed in Table 16.

Table 16: Sodium dodecyl sulfate cleaning

Label	Mass (g)	Concentration (wt%)
DA	2	0.5714
DB	4	1.1429
DC	6	1.7143
DE	8	2.2857
DF	10	2.8571

Ethanol concentration in the range listed in Table 17 was used to confirm that chemicals chosen in this project are feasible alternatives to state of the art technology.

Table 17: Higher concentration ethanol cleaning

Label	Volume (mL)	Concentration (% v/v)
ED	50	14.29
EE	100	28.57
EF	150	42.86
EG	200	57.14
EH	250	71.43
EI	300	85.71

#### 3.4.3 Lab-Scale Comminution

The comminution configurations were Kenwood FP920 horizontal double knife unit in the processor bowl, alternating angled 4-blade unit in the blender jug, and a dry mill with blades similar to the blender. All three configurations were tested for 1 h residence time at three speed settings, except the test using dry mill, which was for 10 seconds.

#### 3.4.4 Large Scale Decontamination and Cleaning

2.5 kg batches of feathers were decontaminated using 2 stages of 0.1485% NaOCl adjusted to pH 10.0 with 1 M NaOH in 25 L water mixed at 10 Hz by a Lamort pulper for 30 minutes. Decontaminated feather was cleaned for 3 stages using 0.15% H<sub>2</sub>O<sub>2</sub> in 25 L water mixed at the same conditions as decontamination. 10 g samples were taken after each step for tests listed in Table 18.

Table 18: Large scale treatment analysis

Time		Analysis							
(h)	Treatment	Bacteria	Colour	Optical Microscope	SEM				
0	Untreated	X	X	X	X				
0.5	1 stage NaOCl		X	X					
1	2 stage NaOCl	X	X	X	X				
1.5	1 stage H <sub>2</sub> O <sub>2</sub>		X	X					
2	2 stage H <sub>2</sub> O <sub>2</sub>		X	X					
2.5	3 stage H <sub>2</sub> O <sub>2</sub>	X	X	X	X				

### 3.4.5 Large Scale Comminution

Two experiments were performed: A) 1.33 kg dry feather in 300 L water processed for 3 hours circulating at 100 m<sup>3</sup> h<sup>-1</sup>. B) 0.947 kg dry feather in 300L water processed for 4 hours circulating at 100 m<sup>3</sup> h<sup>-1</sup>.



4 Results and Discussion

## 4 Results and Discussion

Unprocessed feather appeared straw-like and the barbs were stuck to the rachis in a greasy tangle (Figure 23). Raw wet feather turned brown after 2 days if left at room temperature and had a distinct putrid smell. After washing in regular tap water feathers had their barbs fanned out, but a significant odour remained. Water holding the feather turned black after a few days from bacterial decomposition.



Figure 23: Raw feather. A – Rachis with lipids. B – Tip with brown impurities.

#### 4.1 Lab-scale Decontamination

The main contaminants on the surface of feathers are preen oil, offal fat and faecal matter. Initially, full strength Janola (4% w/v NaOCl) was used to decontaminate unprocessed feather, however, the feathers completely degraded in less than a minute. Feathers treated with diluted Janola containing 1.05% NaOCl did not turn brown or retained their unpleasant odour.

Sodium hypochlorite (NaOCl), the sodium salt of hypochlorous acid, caused lipid bilayer of bacterial cell membranes to lyse. Hypochlorous acid hydrolyses unsaturated fatty acids by adding chlorine and a hydroxyl group to carbonyls. At high concentrations, hypochlorous acid oxidises sulfhydryl groups on cysteine to denature keratin and form hydrochloric acid (HCl). NaOCl reacts with HCl to form chlorine gas. This was prevented by maintaining a pH 10.0 suspension by adding sodium hydroxide (NaOH). The effluent must be thoroughly rinsed away because NaOH degrades fibre and NaOCl releases oxygen violently on contact with hydrogen peroxide.

Soxhlet extraction was used to determine residual fat content, after treating feather using different concentrations of NaOCl (Table A 10). Feather treated with 0.21%

w/v Janola unexpectedly caused final specimen weight to increase; while a small degree of impurities was removed using a concentration ranging from 0.525 to 1.25% w/v. A sample of feather treated using 0.525% Janola is shown in Figure 24. Consequently, 15% sodium hypochlorite diluted to 1% w/v was used to decontaminate feather in large scale processing.



Figure 24: Feather treated by 0.525% Janola for 5 minutes.

## 4.2 Lab-scale Feather Processing

Candidate washing solutions representative of solvents suggested by patents were trialled; these were ethanol, Tween-80, sodium dodecyl sulfate (SDS) and hydrogen peroxide. In these experiments, cleaning and feather cutting were trialled as a combined process using the lab scale blenders described in the previous chapter.

Initially, a Breville BFP30 processor, shown in Figure 11B, was used to mix the feather suspensions, but the rachis tangled around the rotor and blade, causing undue resistance to the motor. In an effort to resolve this, raw feathers were precut to 15-25 mm using scissors. The processor could only be used for 5 min at a time to prevent the motor overheating and intermittent mixing meant the feather settled or floated, resulting in inadequate contact with the cleaning solution. Since feather were pre-cut with scissors before using the blender-processors, it was impossible to determine whether feather was further reduced in size during processing.

At low agitation speed and a feather to liquid ratio of 1:50, feather floated and swirled around with little contact with the blades. Adjusting to 1:1 feather to liquid ratio caused long flight feathers to wrap around the blade causing equipment failure.

Using a blender with different height to diameter ratio and spike blades at the base (Figure 11C) was capable of shredding feather at 1:1 feather to liquid ratio. At low solid concentrations, the suspension spun from the middle to form a vortex and travelled up the side of the container. At high solid concentrations, long rachis wrapped around the blades after a few minutes. A slurry formed, where down feather was reduce in size, but long flight feathers interfered with the rotating blade.

The wider bowl (Figure 11F in page 32) could accommodate the occasional long rachis as the blade was located at the base of the rotor causing less vortex formation. However, agitation caused feather to align parallel to the flat blades so cutting was limited to vertically oriented feathers. The Kenwood FP920 (Figure 11D) as capable of superior mixing, its blades were inefficient in reducing feather into fibre.

The dry mill in Figure 11E had the same shaped blades as the blender. It readily pulverised dry feather but produced a significant amount of powder and only 1 g of completely dry feather could be processed at one time.

Feather cleaned by up to 10% v/v ethanol was generally found to be ineffective and resulted in fluffy light brown feather with an unpleasant odour. Tween-80 caused excessive foaming, and can only be used with gentle mixing. Hydrogen peroxide imparted a sickly sweet bleach odour to the feather. SDS imparted a soap smell to the feather and ethanol imparted a perfume-like odour to washed feather. It was found that feather had to be properly rinsed to remove residual cleaning chemicals and stop further chemical degradation that lead to foreign odour development in the product.

## 4.3 Lab-scale Analysis

#### 4.3.1 Impurity Evaluation

The six-unit Boltac mixer was used to agitate 10 g feather suspended in 500 mL cleaning solutions at 100 rpm for 1 h. The impurities expressed as *n*-hexane extractable content were plotted against concentration in Figure 25 to Figure 27.

It can be seen from Figure 25 that ethanol cleaning reduced the hexane soluble content from about 15% in raw feathers, to about 10% using 80 % ethanol. This

would suggest that a single equilibrium stage would be insufficient in reducing hexane extractable impurities to an acceptable level. Assuming that one third impurities are removed in single stage, it would require at least 3 cleaning stages to produce fibre containing approximately 1% impurities. It was also found that using more than 80 % ethanol resulted in no additional benefit.

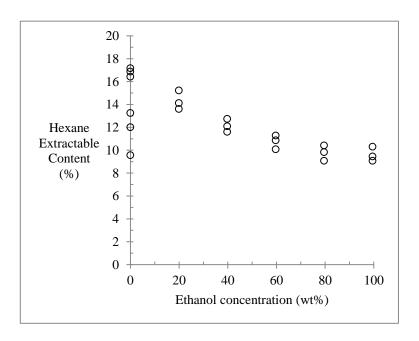


Figure 25: Hexane extractable content after ethanol cleaning.

Using hydrogen peroxide at a concentration ranging from 0.15-0.55% w/v gave similar results to that of ethanol and cleaning efficiency levelled off after 0.25% hydrogen peroxide. Impurity content (Figure 26) also remained above 10% and would therefore require more than one equilibrium stage

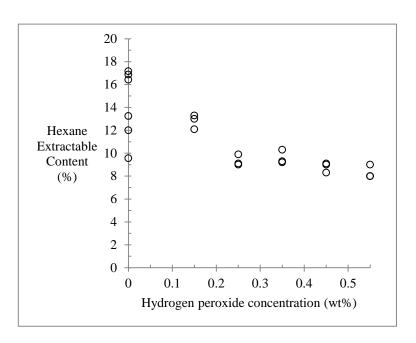


Figure 26: Hexane extractable content after hydrogen peroxide cleaning.

From Figure 27 it can be seen that using SDS at concentrations from  $4 \times 10^{-4}$  to  $2 \times 10^{-3}$  % was ineffective at reducing hexane extractable impurities. It was thought that SDS encapsulated lipids and keratin as micelles but did not separate the two. Another factor may also be that it was more difficult for this amount of SDS to interact with oil adsorbed into feather barbs or rachis and would therefore require a solvent that can easily penetrate the rachis or barbs.

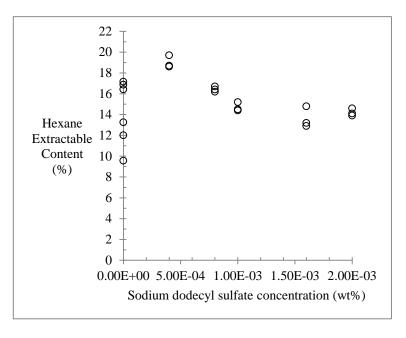


Figure 27: Hexane extractable content after SDS cleaning.

From these results, it was concluded that equilibrium quickly sets in between the hexane extractables in the feather and the amount that can be dissolved in

solution. It would therefore be required to use more than one equilibrium stage to obtain a feather product with very low extractable content.

It was found that hydrogen peroxide would be a better choice of cleaning agent, based on price and safety concern dealing with a solvent. Ethanol has to be recovered using distillation whereas hydrogen peroxide safely decomposes and can be discarded. In large scale experiments, three equilibrium stages using hydrogen peroxide was implemented. Considering the relative insensitivity of impurity removal to hydrogen peroxide removal, it was decided to use 0.15% in large scale trials.

#### 4.3.2 Colourimetry

Feather whiteness after treatment with various concentrations NaOCl is presented in Figure 28. Feathers were whitened to at least 80% after treatment. For concentrations 0.525 and 1.05% equivalent NaOCl, feathers treated for 5 minutes were somewhat whiter than those treated for 10 minutes. Table 19 illustrates the colour of treated feathers using sodium hypochlorite.

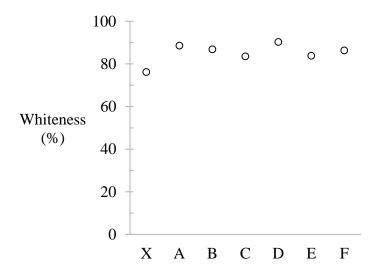


Figure 28: Feather whiteness after Janola treatment. X – Untreated. A – 0.21% NaOCl for 10 minutes. B – 0.21% NaOCl for 5 minutes. C – 0.525% NaOCl for 10 minutes. D – 0.525% NaOCl for 5 minutes. E – 1.05% NaOCl for 10 minutes. E – 1.05% NaOCl for 5 minutes.

Table 19: Feather colour after Janola decontamination.

NaOCl	Time	Average Colour	Whiteness

(%)	(min)	L*	a*	b*	R	G	В	(%)
0	0	78.5	1.4	11.0	207	203	172	76.13
0.21	10	89.5	0.4	8.8	235	236	207	88.57
0.21	5	88.0	0.6	8.8	231	231	202	86.85
0.525	10	85.1	0.7	9.6	224	222	193	83.56
0.525	5	90.9	-0.1	7.1	237	240	214	90.28
1.05	10	85.4	1.7	11.3	228	223	191	83.84
1.05	5	87.8	1.0	11.1	233	230	197	86.34

Feather whiteness after ethanol treatment is presented in Figure 29. Whiteness values were also above 80%. Ethanol concentrations above 14 % did not produce increasingly whiter feathers. Table 20 shows 14% ethanol was most effective at removing colour.

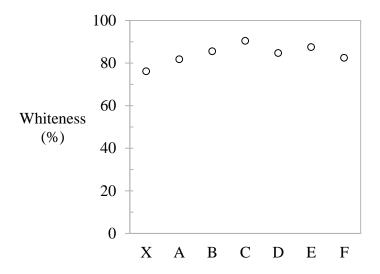


Figure 29: Feather whiteness after ethanol treatment. X – Untreated. A – 1%. B – 3%. C – 14%. D – 42%. E – 57%. F – 85%.

Table 20: Feather colour after ethanol cleaning.

Ethanol	Average Colour	Whiteness

Concentration (%)	L*	a*	b*	R	G	В	(%)
0	78.5	1.4	11.0	207	203	172	76.13
1	83.6	1.0	10.6	221	218	187	81.78
3	86.6	0.5	7.8	227	227	201	85.52
14	90.9	-0.2	6.6	236	240	215	90.37
42	85.8	-0.3	5.8	221	225	202	84.70
57	88.4	-0.1	6.9	230	233	207	87.48
85	83.8	-0.2	6.8	216	219	195	82.43

Feather whiteness values for two mixing methods were compared in the first part of Figure 30. Feathers were suspended in 500 mL water and mixed using the Kenwood processor and the Boltac mixer at 60 rpm. There was no significant difference in whiteness. Feather treatment with ethanol, hydrogen peroxide and sodium dodecyl sulfate are also presented in Figure 30. SDS produced the whitest feathers. Feather cleaned by 500 mL 0.25% H<sub>2</sub>O<sub>2</sub> agitated at 60 rpm for 1 hour in the Boltac was also able to whiten feather to about 90% whiteness. Table 21 shows feather colour removal by mixing equipment and treatment with different chemicals.

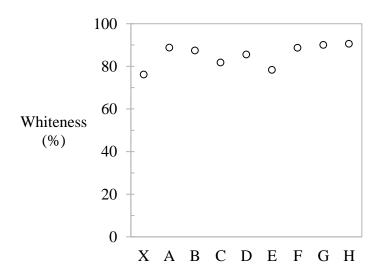


Figure 30: Feather whiteness comparison after mixing and treatment. X – Untreated A – Suspended in water and mixed by processor B – Suspended in water and mixed by Boltac at 60 rpm. C – 1% ethanol. D – 3% ethanol. E – 0.15% H<sub>2</sub>O<sub>2</sub>. F – 0.25% H<sub>2</sub>O<sub>2</sub>. G – 2.28 wt% SDS. H – 2.86 wt%.

Table 21: Feather colour after mixing and treatment.

Treatment		A	verage	Whiteness			
	L*	a*	b*	R	G	В	(%)
No Treatment	78.5	1.4	11.0	207	203	172	76.13
Kenwood Processor	89.6	-0.3	7.3	233	236	210	88.76
Boltac at 60 rpm	88.2	-0.4	5.8	228	232	209	87.44
1% ethanol	83.6	1.0	10.6	221	218	187	81.78
3% ethanol	86.6	0.5	7.8	227	227	201	85.52
0.15% H <sub>2</sub> O <sub>2</sub>	80.4	0.2	9.5	209	209	180	78.30
0.25% H <sub>2</sub> O <sub>2</sub>	89.4	-0.3	6.3	232	236	211	88.70
SDS 2.28 wt%	90.6	0.1	7.3	237	239	213	90.02
SDS 2.86 wt%	91.0	-0.2	5.9	236	241	216	90.58

## 4.3.3 Morphology

Feather decontaminated using 0.21% Janola for 5 minutes and 10 minutes were examined and the results are present in Figure 31. Feather processed for 10 minutes were more yellow than those processed for 5 minutes. Mixing in diluted Janola caused feathers to unfurl improving impurity removal during further treatment.

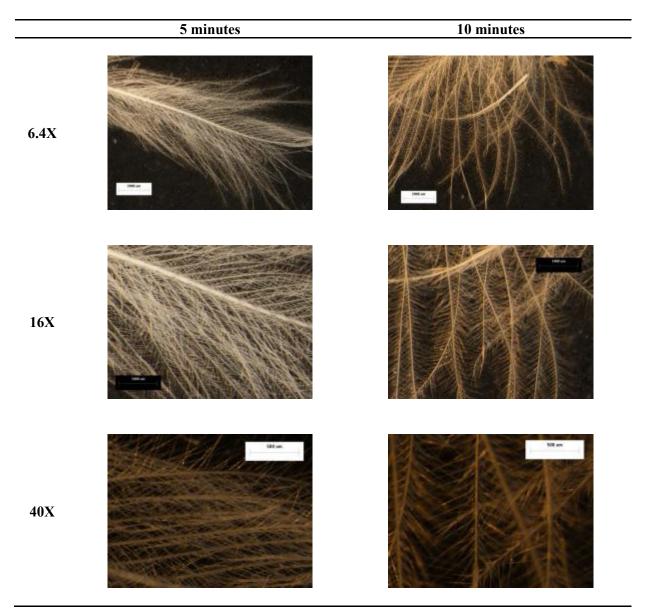


Figure 31: Feather morphology after 5 min or 10 min Janola treatment.

Feather cleaned by 1% and 3% ethanol were compared under optical microscopy (Figure 32). Intricate substructures were clearly visible after the 3% ethanol treatment. The barbs were parallel and barbules were aligned. The barbules were still sticky, presumably as a result of residual lipids after 1% ethanol.

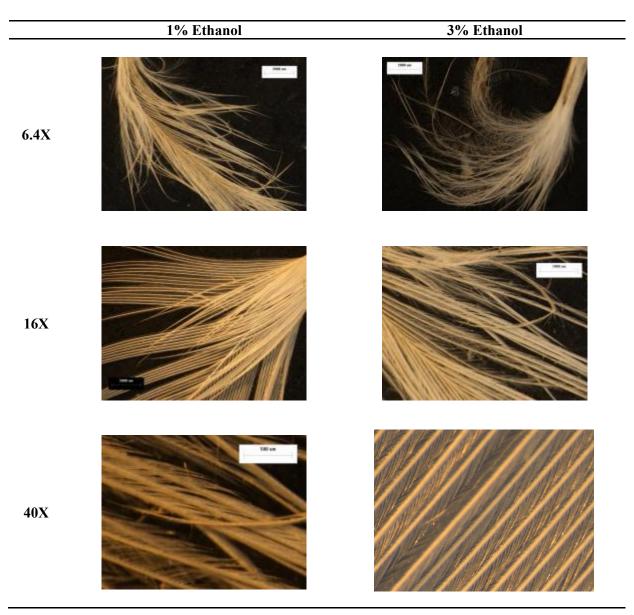


Figure 32: Feather morphology after ethanol lab-scale treatment.

Feather cleaned by 0.15 and 0.25% hydrogen peroxide in the Breville mixer were compared under an optical microscope (Figure 33). In these images, feather fibres are clearly visible. These fine fibres have unfolded away from clumps. Some layers of lipids have been removed to reveal the white surface of feather keratin.

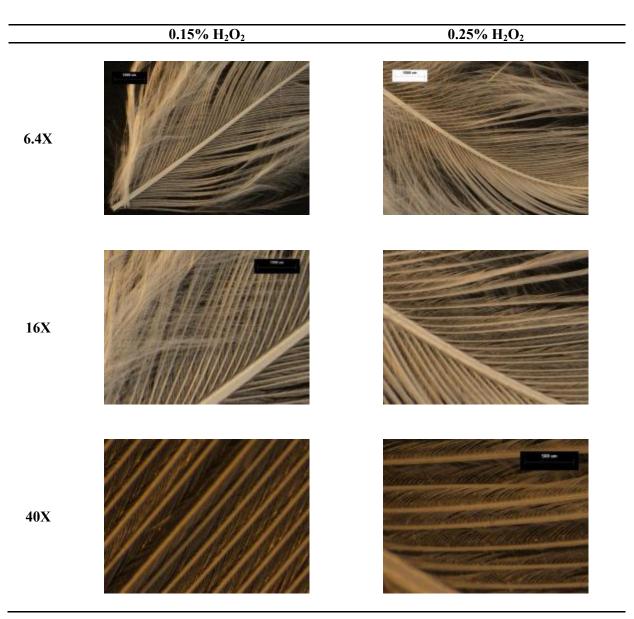


Figure 33: Feather morphology after hydrogen peroxide lab-scale treatment.

From the results presented here it can be concluded that feather treatment can improve colour to 90% whiteness. Colour improvement using ethanol was best at 14.29%, but using concentrated ethanol only 80% whiteness could be reached. SDS produced the whitest feathers, and 0.25% hydrogen peroxide was able to whiten feather to a comparable degree. It was also confirmed that mixing using the Boltac at 60 rpm was just as effective as mixing with Kenwood processor in terms of removing colour.

# 4.4 Large Scale Treatment

Initial large scale decontamination used 1% sodium hypochlorite. Excessive chlorine gas was generated at this concentration.

The chemical equilibrium was upset by acidic pH of the feather. The suspension must remain alkaline for effective decontamination so sodium hydroxide was added in subsequent experiment to increase pH to 10.0.

The suspension became saturated with impurities in 30 minutes when the mixing speed was set at 10 Hz. The liquid fraction was drained and refilled with 25 L water and 250 mL 15% NaOCl adjusted to pH 10.0, or 0.1485% NaOCl. Bacteriological test carried out by NZ Labs found that two decontamination stages at this concentration were adequate. Sodium hypochlorite solution was rinsed after decontamination to prevent further chemical degradation.

The decontaminated feather was suspended in 25 L water and 125 mL of 30% hydrogen peroxide for 30 minutes. The murky liquid fraction was drained. Feather whiteness by colourimetry suggested three stages of 125 mL 13%  $H_2O_2$  in 25 L water 0.15%  $H_2O_2$  sufficiently removed colour from the feather. The wet feather appeared white to visual inspection. The barbs and barbules were fully spread out from the rachis. The feather no longer wet rancid during storage.

#### 4.4.1 Bacteriological Test

The raw feather tested positive for *C. jejuni*. No *Salmonella* was detected in any sample. *Enterobacteriaceae* decreased by a thousand fold after decontamination and was eliminated after cleaning, as shown in Table 22.

Table 22:	Bacterial	test re	sults f	or f	feather	treatment.

Sample ID	Untreated Feather	Decontaminated Feather	Cleaned Feather	
Salmonellae	Not	Not	Not	
in 100 mL	Detected	Detected	Detected	
Campylobacter	C. jejuni	Not	Not	
in 100 mL	Detected	Detected	Detected	
<i>Enterobacteriaceae</i> cfu per mL	20,000	23	<1	

## 4.4.2 Colourimetry

Feather colours were measured and converted to percentage whiteness shown in Figure 34. Average whiteness increased after each decontamination and cleaning stage. Sodium hypochlorite did not remove pigments, while the third stage of hydrogen peroxide affected noticeable change. Table 23 shows feather colour after each treatment step.

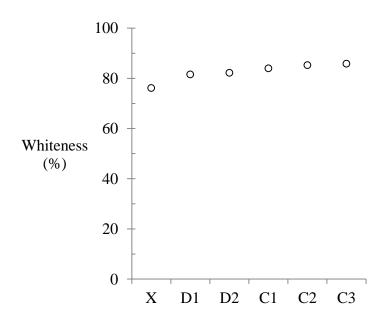


Figure 34: Large scale treatment average feather whiteness. X – Untreated. D1 – 1 stage NaOCl decontamination. D2 – 2 stage NaOCl decontamination. C1 – 1 stage  $H_2O_2$  cleaning. C2 – 2 stage  $H_2O_2$  cleaning. C3 – 3 stage  $H_2O_2$  cleaning.

Table 23: Feather colour after decontamination and cleaning steps.

Tuestment		Whiteness					
Treatment	L*	a*	b*	R	G	В	(%)
No Treatment	78.5	1.4	11.0	207	203	172	76.13
1 stage NaOCl	83.7	0.8	12.6	222	218	184	81.57
2 stage NaOCl	84.3	1.2	12.8	225	219	185	82.19
1 stage H <sub>2</sub> O <sub>2</sub>	85.9	0.5	12.0	227	225	190	83.98
2 stage H <sub>2</sub> O <sub>2</sub>	86.9	0.4	11.2	230	228	195	85.26
3 stage H <sub>2</sub> O <sub>2</sub>	87.4	0.7	11.9	232	229	195	85.79

The large variation was due to the biological nature of the sample. Feather colour varies due to the individual nature of the animal and how it was processed. Different aged individuals has varying amount of preen oil in their feather, where the fatty acid composition contributes to the yellowness. Particulates from dust bathing, pieces of feed and faecal matter may also be present, resulting in a heterogeneous mixture. The untreated feather may have been harvested several hours before decontamination, where bacteria have started to excrete waste material that cause feather to darken. The untreated feather also contains blood, which look pink just after collection but turns brown as it dries. The method of

transporting the feather, such as by flume water, may affect amount of impurities. If the fluids were warm, the pigment from blood may set and become hard to remove.

Table 24 shows feather colour after mixing with Lamort pulper. Both types of impeller achieved about 88% whiteness after 10 minutes. It was difficult to drain the suspension while using a spiral impeller so the disc impeller was used for subsequent large scale treatments.

Table 24: Feather colour after mixing with Lamort pulper impellers.

Impallar	Speed	Time	Average Colour							Whiteness
Impeller S	Speed	(min)	L*	a*	b*	R	G	В		(%)
None	0	0	78.5	1.4	11.0	207	203	172		76.13
Disc	Fast	10	89.4	0.0	7.1	233	236	210		88.64
Disc	Slow	10	89.2	0.0	7.0	232	235	209		88.37
Spiral	Fast	10	90.6	0.0	5.8	235	239	215		90.15
Spiral	Slow	10	88.7	0.0	7.1	231	233	207		87.81
Spiral	Fast	5	89.3	0.1	7.0	232	235	209		88.50
Spiral	Slow	5	84.5	0.3	7.5	220	221	195		83.11

## 4.4.3 Macroscopic Morphology

Dried feather ranged from pale yellow for untreated feather to off white for decontaminated and cleaned feather. The bulk of the clean feather became fluffy and tufts of white down fibre have a soft texture. The flight feather barbs with a waxy waterproof surface reflected light. The dirty feather was in a clumpy, lank and stringy form with debris due to remaining lipids. The decontaminated feather started to unfurl. Each successive stages of cleaning made the feather whiter and the substructures spread out.



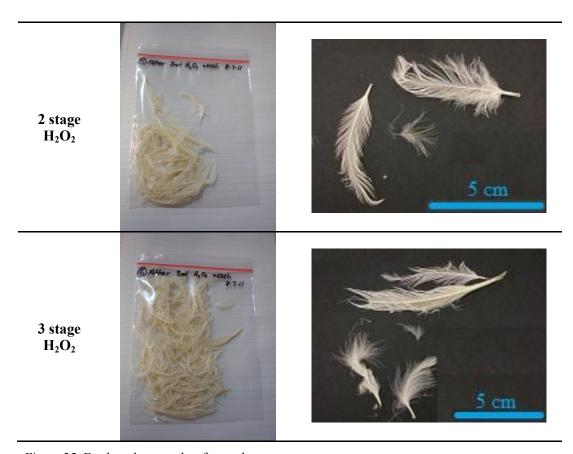


Figure 35: Feather photographs after each treatment step.

#### 4.4.4 Stereoscopic Morphology

Feather structure and form were examined under an optical microscope. Two pieces of untreated feathers are shown in Figure 36. The entire feather is tangled and curled into a clump. Figure 36E shows flecks of yellow debris. Figure 36E and Figure 36F show bundles of fine fibre.

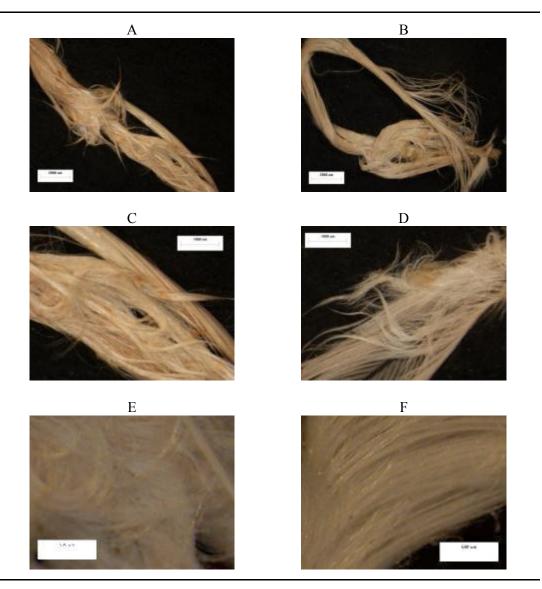


Figure 36: Morphology of untreated feather.

Figure 37 shows two feathers after the first stage of decontamination. The outer edges of the barbs in Figure 37B fold inwards. Figure 37D shows a relatively clean rachis. On the left column Figure 37C and Figure 37E show barbs and barbules still coated with lipids.

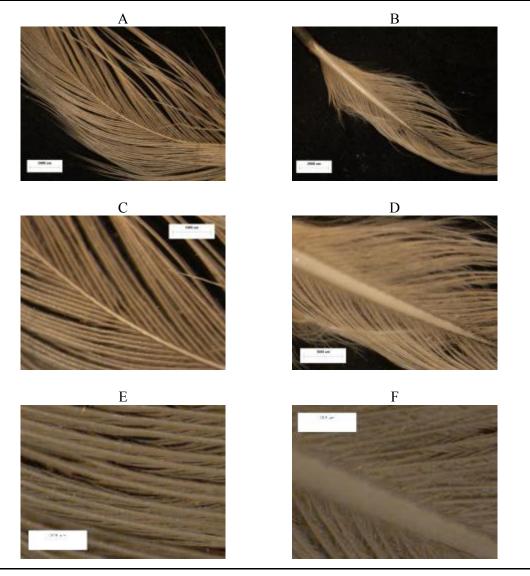


Figure 37: morphology after first stage decontamination.

Figure 38 shows two decontaminated feathers after 2 stages of NaOCl. Both are still folded due to lipid residues. Figure 38E and Figure 38F show pieces of yellow debris coating the barbs and the tip of the feathers.

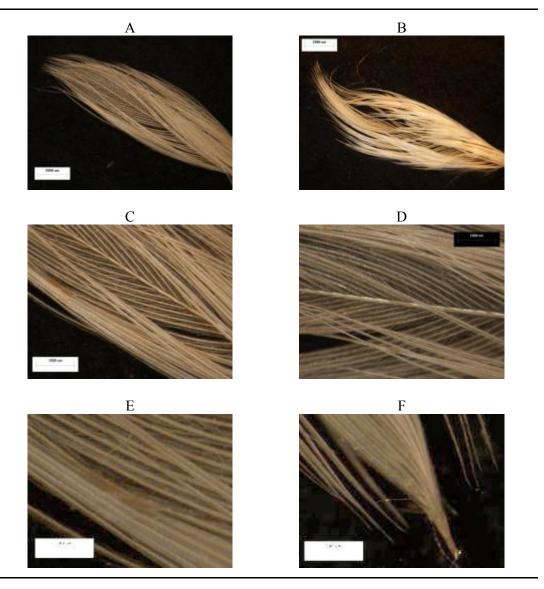


Figure 38: Morphology after second stage decontamination.

Figure 39 shows two feathers cleaned by 1 stage of hydrogen peroxide. Figure 39E and Figure 39F indicate nodular structures are intact.

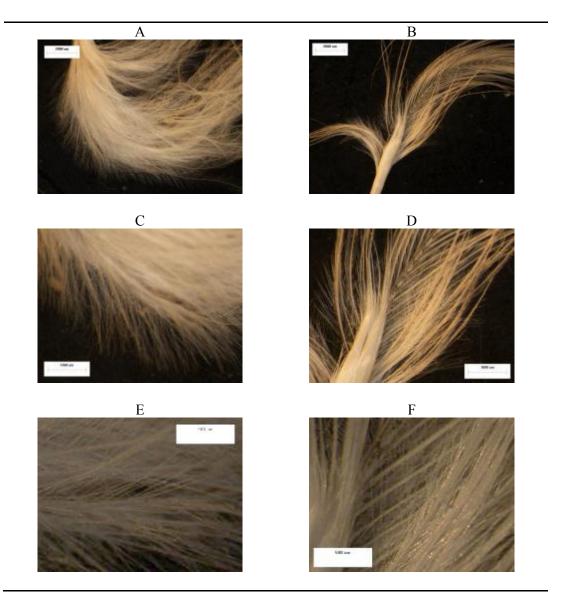


Figure 39: Morphology after first stage cleaning.

Figure 40A has a lot of fine down fibre, while Figure 40B has ordered barbs and barbules, but a large piece of debris where the barb is folded over.

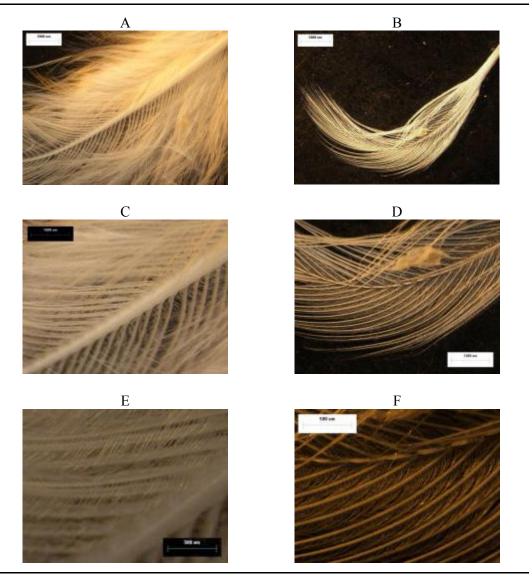


Figure 40: Morphology second stage cleaning.

Figure 41 shows two feathers after 3 stages of hydrogen peroxide cleaning. Figure 41A and D has tufts of fine fibres on the outer surfaces. The feather in Figure 41B has a thin rachis and slightly folded barbs. On closer inspection, the barbs extending at an angle at even spaces (Figure 41D). The fibres were found to scatter light at 40X magnification (Figure 41E).

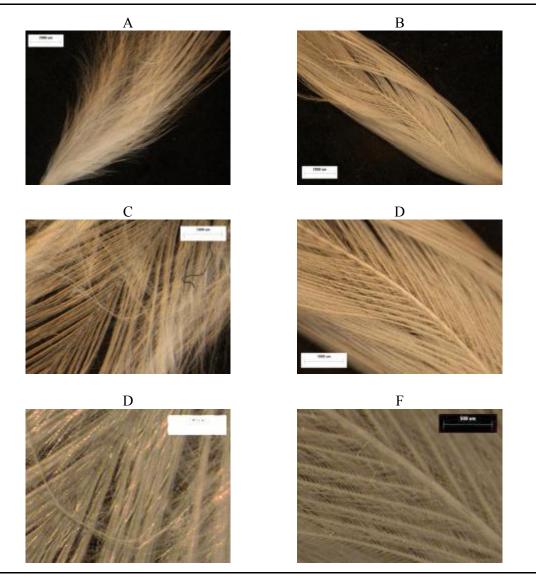


Figure 41: Morphology after third stage cleaning.

#### 4.4.5 Histological Morphology

Figure 42 shows surface morphology for untreated, decontaminated and cleaned feather. Untreated feather Figure 42D had copious debris on the left side and closely linked barbules. Figure 42E in the upper middle section showed hooked barbicels linking to adjacent substructure and a smooth surface in the background. The feather microstructure was not damaged after decontamination, where barbicels at 5  $\mu$ m diameter were found intact. Feathers cleaned by 3 stages of 0.15%  $H_2O_2$  had very few particulate impurities. Figure 42I shows the particles are located between the layer of barbicels and barbules.

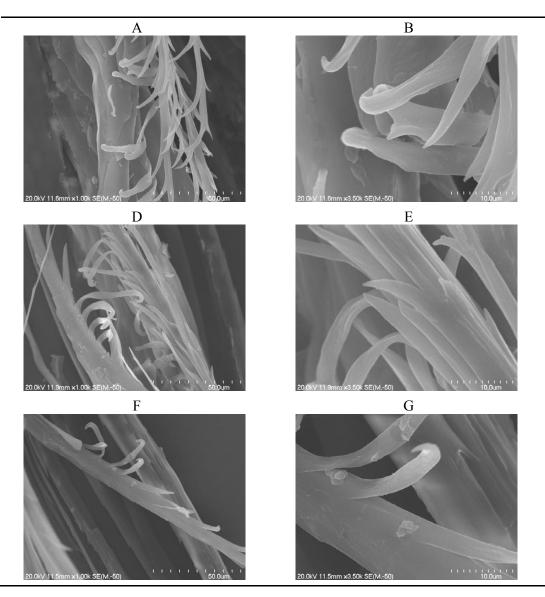


Figure 42: Untreated, decontaminated and clean feather surface morphology.

There were challenges to fully immerse the feather matrix in hydrogen peroxide solution. The curved hollow cylinder of the rachis acted as a capillary. Its base was often connected to dermal fragments, which blocked the entrance to the lipid solute. The barbs, barbules and barbicels were folded together due to the waxy preen oil coat, so lipids must be removed from the exterior to improve wettability before the solvent could gain contact with impurities.

Hydrogen peroxide was used for lipid peroxidation, but not to cause keratin denaturation. Denaturation occurs when disulphide bridges form on cysteine thiol functional groups and the protein aggregates. It was essential to remove the extract from wetted feather because it contains fatty acid radicals, lipid peroxyl radicals, lipid peroxides and cyclic peroxides. The melanin pigments, or

chromatophores, were removed. Feather still appeared off-white to pale yellow due to light diffraction.

## 4.5 Large Scale Comminution

Results from feather comminution are presented in this section. Two comminution experiments were performed, using either 0.44% feather consistency or 0.31%. The pump speed was kept constant at 30 Hz, resulting in a volumetric flow rate of 100 m<sup>3</sup> h<sup>-1</sup>. The first comminution experiment was run for only three hours, and it was thought that allowing an extra hour could increase the yield; the results are shown in Table 25.

Table 25: Comminution mass balance on dry basis.

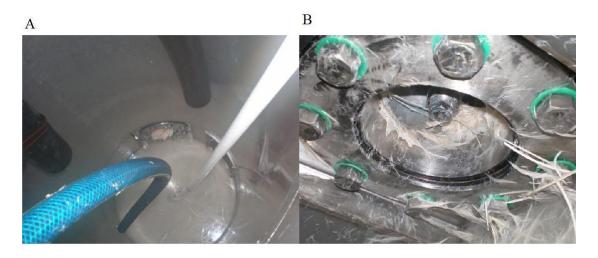
T:		Mass (g)		
Time	Stream	Experiments 1	Experiment 2	
(h)		(0.44% consistency)	(0.31% consistency)	
Total IN	Feather	1325.25	944.47	
End	Fibre	195.16	248.85	
	Feather	1004.26	544.45	
Samples rem	oved			
0	Fibre	0.10	0.07	
	Feather	10.50	7.09	
1	Fibre	2.52	0.26	
	Feather	13.90	3.98	
2	Fibre	0.86	0.25	
	Feather	11.90	2.17	
3	Fibre	2.48	0.27	
	Feather	9.87	3.93	
4	Fibre		0.29	
	Feather		15.2	
Residuals*	Feather	6.43	83.76	
Total feather	· remaining			
Total fibre a	nd sample	200.96	250.138	
Total OUT	_	1296.19	910.7853	
	Yield (%)	14.7	26.3	
	Recovery (%)	97.8	96.4	

A 97.8 % and 96.4 % solids recovery was achieved in experiments 1 and 2 respectively suggesting minimal losses during separation and fibre recovery. About half the mass of feathers is rachis, or there should be a 50 % theoretical fibre yield. The fibre yield for experiment 1 was 14.7%, or 30% of possible fibre was recovered. The second comminution used a lower consistency of 0.31 % and was had an extra hour of operation, leading to a 27 % yield, or 55% of possible fibre recovered. A lower consistency and longer comminution would therefore be preferred for increased fibre recovery.

Solids were allowed to float to the top of the slurry at the end of comminution. Barbs detached from the rachis would agglomerate due to hooked barbicels resulting in highly entangled wet fibres after comminution. This posed significant problems for fibre recovery using gravity filtration. Pouring water over wet feather-fibre mixtures did not produce enough agitation or pressure for filtration. Fibres were mostly curved and angled in a way that exposed barbules and barbicels as hooks. The 1 mm mesh filter would allow vertically oriented particles to pass through, but particles that made contact at an angle were trapped. To overcome this, each batch of feather-fibre mixture was washed several times using the combination of filtration steps as discussed in the previous chapter. However, it is believed that not all fibre recovered and an optimised separation sequence could potentially increase fibre yield further.

After comminution and separation, feather was discovered stuck between the pump impeller and pump chamber (Figure 43); Most of this was uncut rachis. Theses feathers were accounted for as residuals in the mass balance. It was found in subsequent trials that the position of the discharge pipe into the tank strongly influenced the flow pattern in the tank. If adequate mixing is forced by inducing a strong vortex, fibre entrapment in the impeller chamber was also reduced. The chamber was opened to remove all traces of rachis. It would be good practice not to allow rachis to get stuck between impeller blade and chamber because any abrasive material could wear down the impeller and reduce pump efficiency. There was no permanent damage on the impeller shown in Figure 44.

Other problems encountered during processing were feathers floating on the surface and cavitation in the pump. Floating feather meant that they were not circulated through the comminution cycle lading to low fibre yield. This problem was solved by adjusting the position of the discharge pipe to create a vortex dragging feathers down to the suction end of the pump. However, at high pump speeds a significant air would be introduced into the tank leading to cavitation, making the process inefficient.



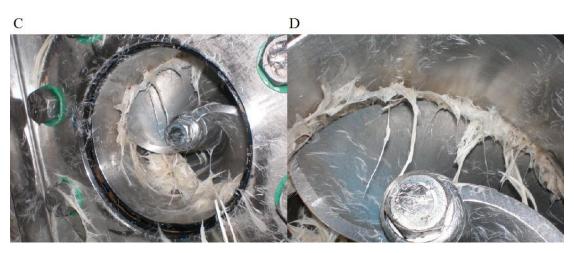


Figure 43: Feather deposition in impeller chamber.



Figure 44: Comminution centrifugal pump impeller.

It was interesting to note that the moisture content of fibre were higher than feather (Table 26). Fibre contained (90  $\pm$  5) % moisture and the uncut feather contained 81  $\pm$  4 %. This indicated that comminution opened voids in the feather by cutting apart the rachis and reducing the amount of hidden surface area between parallel barbs.

Table 26: Comminution moisture content and fibre yield.

		Moisture	Content (%)	)	Fibre Y	ield (%)
Time (h)	Run 1 Fibre	Run 2 Fibre	Run 1 Feather	Run 2 Feather	Run 1	Run 2
0	74.36	96.83	81.69	88.73	0.95	0.98
1	87.99	96.21	79.78	88.08	15.38	6.02
2	90.10	94.23	83.37	90.23	6.77	10.40
3	93.58	95.57	87.11	91.39	20.08	6.36
4		95.36		80.71		1.90

The average fibre yield increased over time for the first experiment, but decreased at hours 3 and 4 during the second experiment. Considering that the second experiment had an overall higher fibre yield, this is a somewhat unexpected result. It was thought that the inherent heterogeneity of the system was highlighted in this result. At any given time a significant amount of feather may be contained in the pipes or pump chamber leading to a feather consistence lower than expected in tank. Ensuring good fibre recovery would imply fairly long processing times to overcome this.

Bulk feather suspension temperature increased over time (Figure 45). Slurry characteristics are expected to be highly dependent on temperature. Bulk slurry viscosity, Reynolds number and head loss were calculated and graphed in Figure A 5 to Figure A 7, respectively. Furthermore, it can be expected that comminution may also be different at higher temperatures. According to literature, only around 1% of energy during comminution is imparted to the material. The rest ends up heating the surrounding medium. It is evident from the large temperature difference between run 1 and 2 and the experiment without feather (Figure 45). Feathers are mostly protein and changing the operating temperature could lead to degradation. It may therefore be required to cool the comminution circuit when done on a large scale, or if longer comminution times are required.

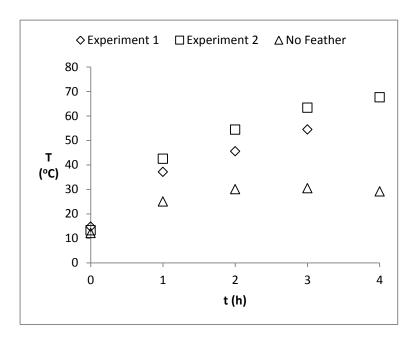


Figure 45: Comminution slurry temperature change through time.

## 4.5.1 Macroscopic Morphology

The fibres and feathers from comminution are displayed in Figure 45.



Figure 46: Dried and packaged fibre and feather from comminution.

Figure 47 shows photographs of fibre and feather fraction sampled at each hour of comminution. The amount of fibre recovered from samples increased over time, however, the feather fraction still had significant amount of entrained fibre.

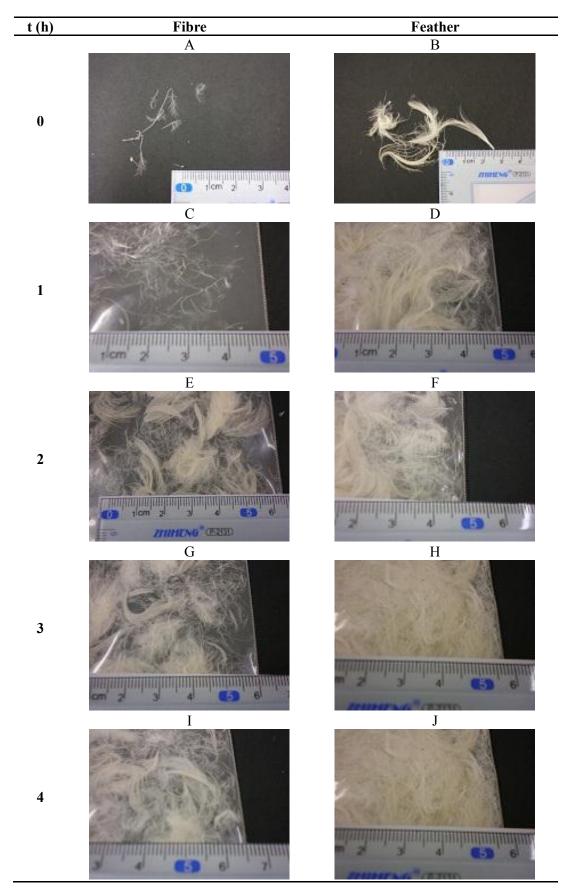


Figure 47: Comminution feather and fibre photographs.

#### 4.5.2 Stereoscopic Morphology

The fibre and feather fractions were examined under an optical microscope. Morphology prior to comminution is shown in Figure 48 and feathers were also filtered before comminution to qualitatively assess the degree to which fibres detached from the rachis after decontamination and cleaning.

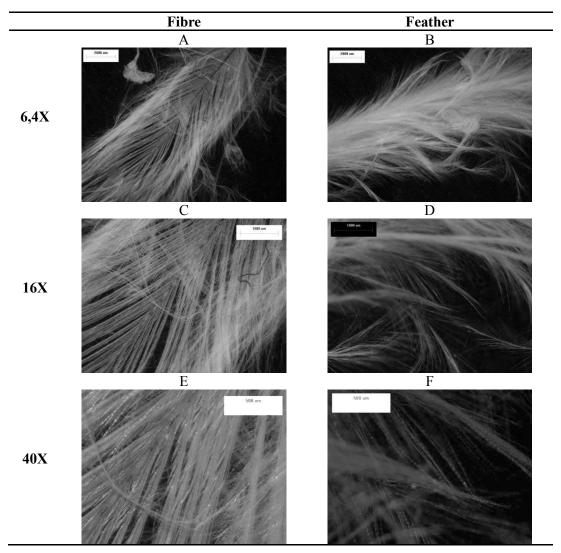


Figure 48: Feather morphology before comminution.

In Figure 5A feather with most of its fibre intact are shown. Figure 48C contains a highly concentrated area of fibre. Figure 48E is the same area, where the nodes are barbules and barbicels. The bundled feather shown in Figure 48B did not go pass through the 1 mm filter. Figure 48D shows that particles in the feather fraction were larger and had more tortuous bends. Nodular substructures were also present in Figure 48F.

Fibre and feather after 1 hour of comminution are shown in Figure 49.

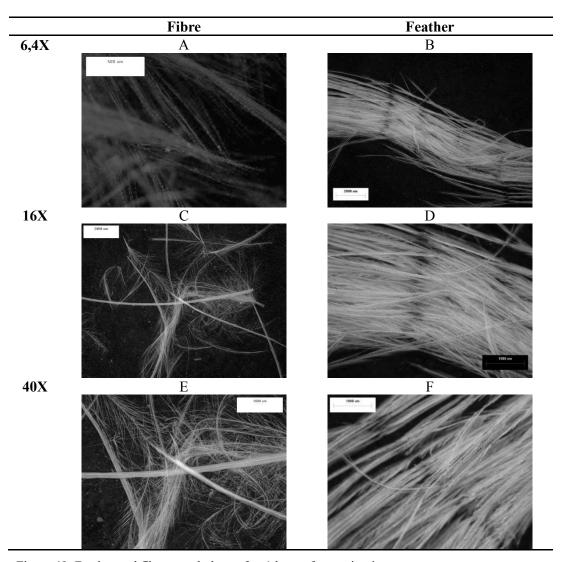


Figure 49: Feather and fibre morphology after 1 hour of comminution.

Fibres in Figure 49A were about 10 mm long and very fine. Short and relatively straight rachis of about 8 mm long passed through the 1 mm filter because its diameter (labelled with arrows in Figure 49E) was only 100  $\mu$ m. Figure 49C showed how similar this group of rachis were compared to the barbs. A bundle of barbs has been partially sheared in B and the faint line down the middle of Figure 49D indicates where the impeller has made contact. From Figure 49F it can be seen that the width of a cut was less than 500  $\mu$ m.

Figure 50 are images of feather and fibre after 3 hours comminution.

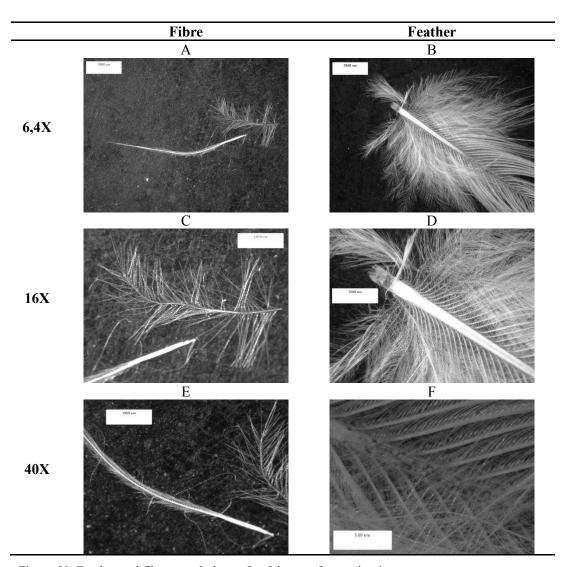


Figure 50: Feather and fibre morphology after 2 hours of comminution.

The centre of Figure 50A shows a sheared rachis, bent in an obtuse angle. In Figure 50C a 2 mm wide barb with a few missing barbules is shown. The 10 mm rachis in Figure 50E has had its barbs sheared to approximately 200  $\mu$ m wide. The 4 mm wide, over 1 cm long semi-plume in Figure 50B still contains a lot of fibre after 2 hours of rigorous cutting. Figure 50D and Figure 50F highlights the durability of rachis against mechanical deformation, still showing highly uniform barbules. The translucent rachis width was 200  $\mu$ m, while the spaces between parallel barbs were 100  $\mu$ m.

Figure 51 are feather and fibre after 3 hours of comminution.

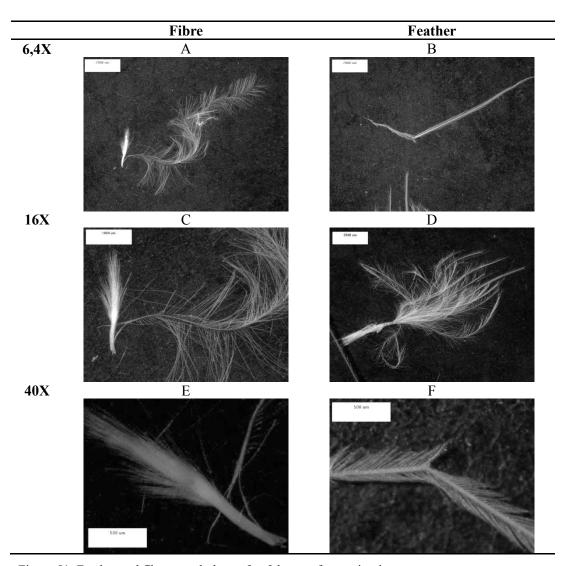


Figure 51: Feather and fibre morphology after 3 hours of comminution.

Figure 51A shows a small 200 µm wide, 2 mm long bundle containing only barbs. The down fibre to its right curls in different directions. Figure 51C shows plenty of nodules. Figure 51E is a bundle showing a wealth of fibre in its early developmental stage. Figure 51B shows a 12 mm rachis bent in an acute angle with barbs sheared off. Figure 51D is a 6 mm wide developing bundle with differentiated barbules with a pinched mid-section and Figure 51F is the bent rachis, where remaining barbs act as hooks to prevent the rachis from passing through the filter.

Fibres after comminution shown in Figure 52 were finer and shorter.

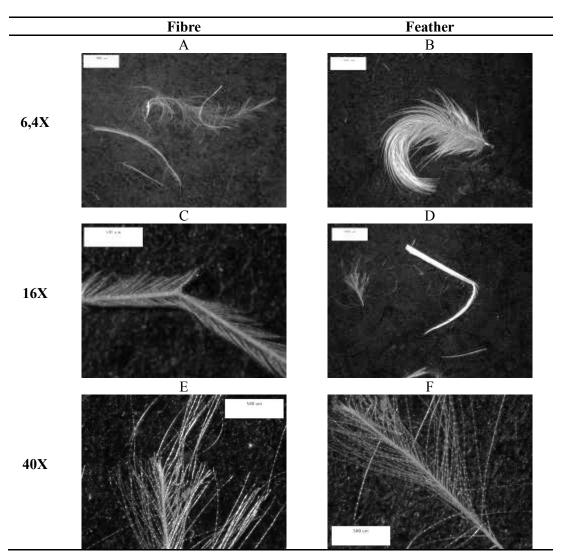


Figure 52: Feather and fibre morphology after 4 hours of comminution.

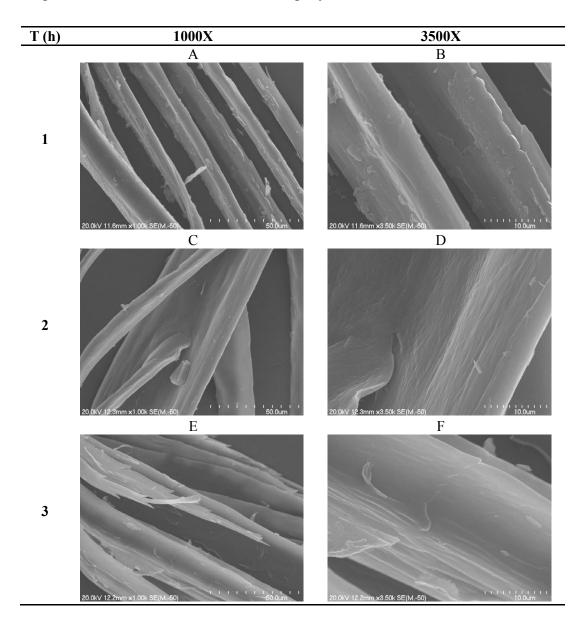
Figure 52A shows a down feather, a thin rachis and barbules. Figure 52C is a barb fibre with a substantial amount of barbules and sub-structural nodes are present in Figure 52E. Feathers such as the one shown in Figure 52B did not get sheared because it was smaller than the impeller clearance. In Figure 52D shows some fibre that should have passed through the filter; the very small piece of down feather remained due to its width. The sickled-shaped rachis was bent at a sharp angle. Figure 52E and F shows the down feather maintained its structural integrity.

From these images it is clear that comminution has reduced average feather size, and that some rachis was recovered with fibres. Also, some usable fibre remained in the feather fraction. Comminution was found to be a process whereby fractions of feather were stripped from larger rachis, as opposed to fracturing distinct feather rachis. Over time, the fibre fraction reduced in size, although qualitative

data was not presented in this section. It is clear that longer comminution times are required to produce a fibre product of reasonable quality.

## 4.5.3 Histological Morphology

Figure 53 shows fibre surface for every hour of comminution. The parallel barbs in Figure 53A have a diameter of 10 um. In Figure 53B, the barbs have chipped edges. After 2 hours, the rachis still had smooth surfaces as shown in Figure 53C and a piece of powder feather remains in the lower middle section of the image. Figure 53D shows where the barbs are slightly torn from the rachis.



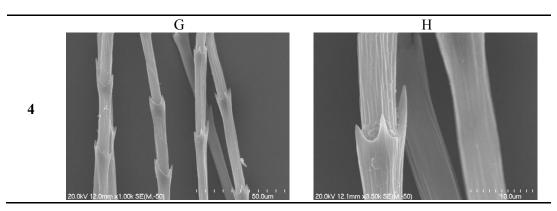


Figure 53: Fibre surface morphology in comminution.

At 3 hours, Figure 53E shows a bundle of barbs and Figure 53F is the barb with peeled layers. After 4 hours of comminution, the barbules in Figure 53G showed serrated barbicels. The barbule in Figure 53H had surface striations. This artefact is unlikely to be from pump impeller shear, but may be the effect of drying.

Many stages of filtration in series were necessary, which indicate continuous filtration would result in better separation. The proposed method is to cycle the suspension through a pressure screen controlled by valves that thickens the fibre fraction. A process diagram was devised to summarise decontamination, cleaning, comminution, filtration and drying Figure A 8.

#### 5 Conclusions

Raw feather supplied by Wallace Corporation contained about 15% hexane extractables such as preen oil, as well as other contaminants such as offal, faecal matter and process water. Treating feather with sodium dodecyl sulphate solutions did not reduce the hexane extractable content, while high concentration ethanol or hydrogen peroxide solutions were able to reduce hexane extractables to about 10% in one equilibrium stage.

Large-scale feather treatment required 2 equilibrium stages for decontamination using sodium hypochlorite and 3 stages for cleaning. Each equilibrium stage contained 25 L solution at a concentration of 0.1485% sodium hypochlorite adjusted to pH 10.0 with 1 M sodium hydroxide for decontamination and 0.15% hydrogen peroxide for cleaning. A Lamort pulper with a disc impellor was used to mix the feather suspension at 10 Hz for 30 minutes in each stage.

Bacteriological testing found no *Salmonella*, *Campylobacter* or *Enterobacteriaceae* on treated feather. Using colour analysis it was revealed that raw feather showed 76% whiteness, which was increased to 86% whiteness after three stages hydrogen peroxide washing. Clean feathers were fluffy and off-white, compared to a brownish yellow colour of raw feather with a foul odour.

Clean feather was comminuted in a stainless steel tank containing 300 L water using a centrifugal pump at 30 Hz impeller speed under full recycle for up to 4 hours. At the end of the process partially cut feather was separated from fibre using a series of gravity filters and dried.

Half of the mass of feather is rachis, so the theoretical fibre yield after comminution was 50%. It was found that the yield after three hours comminution was 15% and increased to 27% after four hours. Morphological studies revealed that the fibre surface was not damaged by this comminution method. Fibre product consists of barbs with intact barbules and thin rachis with barbs sheared off.

#### 5.1 Recommendations

The large scale process could be improved to produce fibre quality that is more consistent with that obtained in lab scale processing, in terms of fibre whiteness and hexane extractable content.

Information on fibre diameter and length distribution at different operating conditions would be helpful for optimising pump impeller selection as well as pipes and valve arrangement that would reduce cavitation and fibre deposition.

The recovery process should use continuous filtration, such as the Beloit pressure screen.

Production scale development should consider equipment available within the rendering industry and can be supplemented with those available from the pulp and paper industry. These could include continuous filtration equipment, such as a Beloit pressure screen.

Further testing such as single fibre tensile tests would allow comparison with other types of fibre. Lastly, the product should be tested in applications such as insulating materials and composites to determine its suitability.

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Appendices

# Appendices

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# **Appendix 1: Chicken Feather**

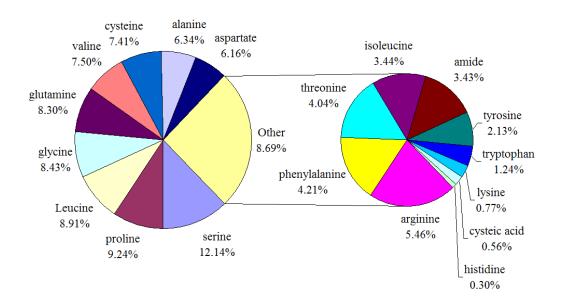


Figure A 1: Feather rachis amino acid mass composition [21].

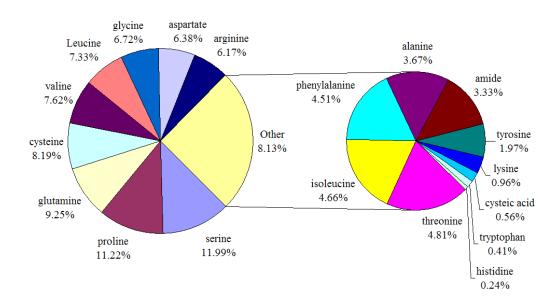


Figure A 2: Feather barbs amino acid mass composition [21].

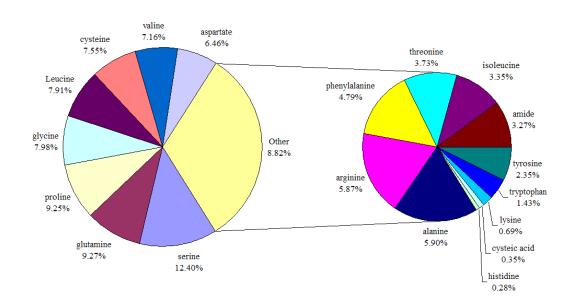


Figure A 3: Feather calamus amino acid mass composition [21].

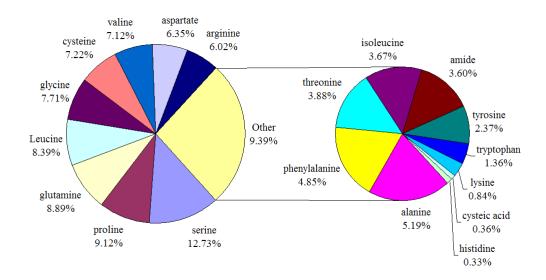


Figure A 4: Feather medulla amino acid mass composition [21].

Table A 1: Rendering equipment manufacturers

Rendering Equipment Manufacturers	Description	
Alfa Laval	Heat transfer, centrifugal separation and fluid handling equipment for the poultry processing plants.	
GEA Westfalia Separator Group (est. 1893)	Mechanical separation products.	
Napier Engineering and Contracting Ltd. (est. 1866) Pacific Terminals NZ	Niven Abattoir equipment for rendering plants.  Handle and store bulk flammable and combustible liquids including chemicals,	
Pinches Consolidated Industries (est. 1947) Rendertech (est. 1988)	edible oils and fats.  Processing and drying technology using superheated steam dryers.  Protein recovery and Rendering, wastewater treatment, odour control and bulk storage	
Transpacific Industries Group (NZ) Ltd (TPI)	tanks. Provides waste and environment services by transporting liquid, hazardous and solid waste to recover resources and manage waste responsibly.	

Table A 2: Company in the poultry by-products industry

Company	Description	
Tradeskins NZ Ltd (est. 1992)	Leather trading company exporting skins, hides,	
	pelts and rendered animal products	
Ritex International Ltd. (est. 1988)	Imports industrial and household textile	
	packaging products	
Kemin Industries NZ Pet food manufacturer		
Mars Petcare New Zealand	Canned, chilled, dry and shelf-stable cat and dog	
	food and treats.	

Table A 3: Waste water bylaw summary [79, 80]

Compound or Test Name	Amount
Solids dimensions	Less than 15 mm
Suspended solids	$2 \text{ kg m}^{-3}$
Settling solids	$50 \text{ mL L}^{-1}$
Biodegradable oil, fat or grease <sup>1</sup>	Less than 500 g m <sup>-3</sup>
Colour <sup>2</sup>	Absorbance less than 0.3010
	Transmissivity greater than 50%
Acidity or alkalinity	pH 6.0 to 10.0
Five-day biochemical oxygen demand (BOD <sub>5</sub> )	Less than 1 kg m <sup>-3</sup>
Methylene Blue Active Substances	300 g m <sup>-3</sup>
Chlorine (Cl <sub>2</sub> )	
Free chlorine	5 g m <sup>-3</sup>
Hypochlorite	5 g m <sup>-3</sup> 10 g m <sup>-3</sup>

1 – When emulsion is stable at 15°C, or the emulsion is diluted by a factor of 10 by raw sewage at pH 4.5 to 10.0

2 – effluent diluted by a factor of 10 with distilled water and tested at 254 nm

# **Appendix 2: Analysis Procedures**

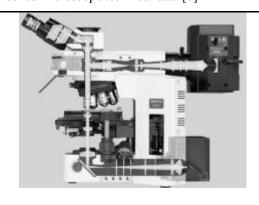
Table A 4: Potential pathogens found on chicken feather.

Microorganisms	Description
Campylobacter	Small, spiral, gram-negative, non-spore-forming rods, cork-screw
[68]	motility with polar flagella, micro-aerophilic, oxidase positive, reduce nitrate
Enterobacteriaceae	Gram-negative, non-spore-forming bacilli, facultative anaerobic,
[89]	ferments glucose, reduce nitrates to nitrites, grows on bile salts media and are oxidase negative
Salmonella	Motile Enterobacteriaceae, catalase positive, oxidase negative
[69]	Biochemistry: H <sub>2</sub> S production, lysine decarboxylation, negative urease reaction, uses citrate as carbon source, ferments glucose to produce gas, no lactose or sucrose fermentation.

Table A 5: Procedure used by NZ Lab for bacteriological test.

Preparation	Weigh, dilute and emulsify or suspend as required; dilutions	
(APHA (4) 2.527)	in 0.1% peptone water	
Campylobacter	Filter 100 mL of sample using 0.5 µm filter. Enrichment in	
MIMM 7.3. 4 <sup>th</sup> Ed	Bolton Broth, incubation at 37°C for 4 h in microaerophilic	
(Modified)	atmosphere, then at 42°C for 44 h. Isolation of mCCda, incubate	
[68]	at 42°C for 48 h in microaerophilic atmosphere. Confirmation by: oxidase, Gram stain, motility, Growth at 37°C, antibiotic sensitivity, hippurate hydrolysis	
Enterobacteriaceae	Count on Petrifilm, incubate at 37°C for 24 h.	
<b>MIMM 8.2</b>		
(Modified) [89]		
Salmonellae	Filter 100 mL of sample using 0.45 µm filter. Resuscitation	
<b>MIMM 7.7</b>	Buffered Peptone water, Enrichment Rappaport-Vassiliadis Soya	
(Modified)	peptone broth, Isolation XLD, Modified Brilliant Green agars,	
[69]	Confirmation using serabact or Entrotube and serological	
	screening. If necessary samples are sent to ESR-CDC Porirua for	
	confirmation. Limits of detection: Not applicable.	
	Presence/absence test.	

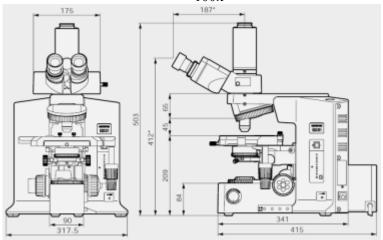
Table A 6: Olympus BX2 series microscope technical data [6]



## Side cross section

Microscope	Optical System	UIS optical system	
frame	Focus	Vertical: 25 mm stage stroke with coarse	
		adjustment limit stopper, torque adjustment	
		for coarse knobs	
		Stage mounting positions with high	

		sensitivity fine focusing knob (minimum adjustment at 1 µm.)	
	Illuminator	Koehler illumination for transmitted light.	
		12V100W halogen bulb, light preset switch,	
		intensity LED indicator and filters	
Observation	Widefield	Binocular inclined 30°, Tilting bionocular	
Tube		inclined 5° – 35°, Trinocular inclined 30° and	
		Ergo binocular inclined 0° – 25°	
	Super Widefield	Trinocular inclined 24°	
Stage		Coaxial, rotational control and torque	
		adjustment	
<b>Condenser</b> Options allow magnification range		Options allow magnification range 1.25x to	
		100x	



Front and Side Line drawings

Table A 7: Konica Minolta CR-410 Chroma Meter Technical Data [7]

Equipment	Chroma Meter and its	<b>Data Processor</b>	
Model	CR-410	Data Processor	DP-400
Illumination	Wide-area	Viewing Angle	$0^{\rm o}$
Light source	Pulsed xenon lamp	Detector	6 Silicone cells
Display range	Y: 0.01 to 160.00%	Battery	800 measurements
	(reflectance)	performance	
Measurement	1 s	Measurement	3 s
time		interval	
Measurement	Φ50	illumination area	Ф53
area			
Colour space	L*a*b*	Illuminant	$C, D_{65}.$
Operating	0 to 40°C	Operating	85% relative humidity
temperature		Humidity	
Observer: 2 degr	ees, closely matches CIE 19	931 standard observers	3
Repeatability: W intervals)	ithin $\Delta E*ab0.7$ standard de	viation (white plate m	easured 30 times at 10 s

Inter-instrument agreement: ΔE\*ab: within 0.8 average of 12 BCRA series II colours

Table A 8: L\*a\*b\* conversion to XYZ and RGB [90].

Step	Formula and Condition
1	$var_Y = (L^* + 16) / 116$
	$var_X = a^* / 500 + var_Y$
	$var_Z = var_Y - b* / 200$

```
2
       if (var_Y^3 > 0.008856) var_Y = var_Y^3
       else var_Y = (var_Y - 16 / 116) / 7.787
       if (var_X^3 > 0.008856) var_X = var_X^3
       else var X = (var X - 16 / 116) / 7.787
       if (var Z^3 > 0.008856) var Z = var Z^3
       else var_Z = (var_Z - 16 / 116) / 7.787
3
       X = ref_X * var_X, //ref_X = 95.047, Observer= 2°, Illuminant= D65
       Y = ref_Y * var_Y, //ref_Y = 100.000
       Z = ref_Z * var_Z, //ref_Z = 108.883
4
       var_X = X / 100, //X from 0 to 95.047, Observer = 2°, Illuminant = D65
       var_Y = Y / 100, //Y \text{ from } 0 \text{ to } 100.000
       var_Z = Z / 100, //Z \text{ from } 0 \text{ to } 108.883
       var_R = var_X * 3.2406 + var_Y * -1.5372 + var_Z * -0.4986
5
       var_G = var_X * -0.9689 + var_Y * 1.8758 + var_Z * 0.0415
       var_B = var_X * 0.0557 + var_Y * -0.2040 + var_Z * 1.0570
6
       if ( var_R > 0.0031308 ) var_R = 1.055 * ( var_R ^ ( 1 / 2.4 ) ) - 0.055
       else var_R = 12.92 * var_R
       if ( var_G > 0.0031308 ) var_G = 1.055 * ( var_G ^ ( 1 / 2.4 ) ) - 0.055
       else var G = 12.92 * var G
       if ( var_B > 0.0031308 ) var_B = 1.055 * ( var_B ^ ( 1 / 2.4 ) ) - 0.055
       else var_B = 12.92 * var_B
7
       R = var_R * 255
       G = var\_G * 255
       B = var_B * 255
```

# **Appendix 3: Lab-scale Treatment**

Table A 9: Lab-scale Soxhlet extraction sample treatment description.

Label	Description	Label	Description
X1	Unprocessed feather	HA	H <sub>2</sub> O <sub>2</sub> 0.35 wt%
X2	Unprocessed feather	HB	$H_2O_2 0.45 \text{ wt}\%$
X3	Unprocessed feather	HC	$H_2O_2 0.55 \text{ wt}\%$
X4	Unprocessed feather	DA	SDS 0.00057 wt%
X5	Unprocessed feather	DB	SDS 0.00114 wt%
X6	Unprocessed feather	DC	SDS 0.00171 wt%
J1	0.21% NaOCl for 5 min	DD	SDS 0.00229 wt%
J2	0.525% NaOCl for 5 min	DE	SDS 0.00286 wt%
J3	1.25% NaOCl for 5 min	E1-3	19.9% ethanol
J4	0.21% NaOCl for 10 min	E4-6	39.8% ethanol
J5	0.525% NaOCl for 10 min	E7-9	59.7% ethanol
J6	1.25% NaOCl for 10 min	E10-12	79.6% ethanol
EA	5 % ethanol	E13-15	99.5% ethanol
EB	7% ethanol	H1-3	$0.15\% \ H_2O_2$
EC	10% ethanol	H4-6	$0.25\%~{\rm H_2O_2}$
ED	14% v/v ethanol	H7-9	$0.35\% \ H_2O_2$
EE	29% v/v ethanol	H10-12	$0.45\% \ H_2O_2$
EF	43% v/v ethanol	H13-15	$0.55\% \text{ H}_2\text{O}_2$
EG	57% v/v ethanol	D1-3	0.0004% SDS
EH	71% v/v ethanol	D4-6	0.0008% SDS
EI	86% v/v ethanol	D7-9	0.0012%SDS
		D10-12	0.0016% SDS
		D13-15	0.002% SDS

Table A 10: Lab-scale Soxhlet extraction calculations.

Label	Mass (g)						Impurity
	Thimble	Feather	Glass Wool	Final Container +Sample	Final	Change	(wt%)
X1	3.212	3.633	0	6.248	3.036	0.597	16.42
<b>X2</b>	3.829	3.335	0	6.763	2.934	0.401	12.01
<b>X3</b>	4.023	4.084	0	7.716	3.693	0.391	9.57
<b>X4</b>	3.229	2.932	0	5.772	2.543	0.388	13.25
X5	4.073	3.080	0	6.625	2.552	0.529	17.16
<b>X6</b>	3.587	3.038	0	6.112	2.525	0.512	16.87
J1	3.44	2.63	0.07	6.29	2.78	-0.15	-5.79
J2	4.40	2.65	0.07	7.11	2.63	0.03	0.97
J3	3.21	2.13	0.06	5.33	2.06	0.07	3.42
J4	3.85	2.61	0.12	7.67	3.69	-1.08	-41.53
J5	4.49	2.40	0.12	6.88	2.27	0.13	5.54
J6	4.04	2.07	0.13	5.92	1.74	0.33	15.73
EA	3.737	3.681	0.131	6.634	2.766	0.915	24.86
EB	4.109	2.965	0.184	6.943	2.651	0.315	10.61
EC	3.516	3.084	0.143	6.229	2.570	0.514	16.65

Label	Mass (g)						Impurity
	Thimble	Feather	Glass Wool	Final Container +Sample	Final	Change	(wt%)
ED	3.045	2.702	0.215	5.620	2.575	0.127	4.69
EE	3.087	2.760	0.233	5.776	2.689	0.071	2.56
EF	3.329	2.394	0.295	5.740	2.412	-0.018	-0.74
EG	3.397	2.449	0.313	6.349	2.953	-0.504	-20.56
EH	3.338	2.713	0.144	5.934	2.596	0.117	4.31
EI	3.630	2.933	0.134	5.902	2.273	0.660	22.50
HA	3.199	3.091	0.157	6.217	2.861	0.230	7.44
HB	3.816	3.058	0.189	6.857	2.851	0.207	6.76
HC	3.267	3.079	0.197	6.194	2.730	0.349	11.33
DA	3.316	2.888	0.093	5.882	2.473	0.415	14.37
DB	3.518	2.919	0.134	6.140	2.488	0.431	14.76
DC	3.495	1.821	1.250	6.146	1.404	0.417	22.90
DD	3.515	2.873	0.247	6.146	2.384	0.489	17.03
DE	3.675	3.241	0.094	7.076	3.307	-0.066	-2.04
<b>E1</b>	3.155	1.823	0.154	4.884	1.575	0.248	13.60
<b>E2</b>	3.309	1.013	0.120	4.299	0.870	0.143	14.12
<b>E3</b>	3.985	1.340	0.883	6.004	1.136	0.204	15.22
E5	3.523	1.860	0.179	5.346	1.644	0.216	11.61
<b>E6</b>	3.346	1.045	0.753	5.011	0.912	0.133	12.73
<b>E4</b>	3.512	2.010	0.160	5.439	1.767	0.243	12.09
<b>E7</b>	3.349	0.655	0.092	4.030	0.589	0.066	10.08
<b>E8</b>	4.013	0.515	0.118	4.590	0.459	0.056	10.87
<b>E9</b>	3.430	1.373	0.331	4.979	1.218	0.155	11.29
E12	3.701	1.630	0.428	5.611	1.482	0.148	9.08
E10	3.311	0.365	0.090	3.728	0.327	0.038	10.41
E11	3.301	1.750	0.172	5.051	1.578	0.172	9.83
E13	3.060	0.508	0.197	3.717	0.460	0.048	9.45
E14	3.202	1.080	0.128	4.312	0.982	0.098	9.07
E15	3.786	2.475	0.820	6.826	2.220	0.255	10.30
H1	3.199	1.830	0.154	4.945	1.592	0.238	13.0
H2	3.759	1.120	0.110	4.853	0.984	0.136	12.1
Н3	4.305	2.767	0.154	6.858	2.399	0.368	13.3
H4	3.607	1.744	0.152	5.330	1.571	0.173	9.9
H5	3.511	1.208	0.190	4.800	1.099	0.109	9.0
H6	3.023	1.979	0.178	5.000	1.799	0.180	9.1
H7	3.379	2.868	0.126	6.108	2.603	0.265	9.2
H8	3.734	1.112	0.166	4.897	0.997	0.115	10.3
Н9	4.132	2.893	0.164	6.919	2.623	0.270	9.3
H10	3.918	0.793	0.114	4.753	0.721	0.072	9.1
H11	3.892	2.696	0.159	6.504	2.453	0.243	9.0
H12	3.493	2.584	0.061	5.924	2.370	0.214	8.3
H13	3.766	1.130	0.163	4.957	1.028	0.102	9.0
H14	3.988	0.923	0.107	4.944	0.849	0.074	8.0

Label	Mass (g)						Impurity
	Thimble	Feather	Glass Wool	Final Container +Sample	Final	Change	(wt%)
H15	3.584	2.764	0.136	6.264	2.544	0.220	8.0
<b>D1</b>	3.259	0.497	0.054	3.717	0.404	0.093	18.7
<b>D2</b>	3.919	1.567	0.058	5.235	1.258	0.309	19.7
<b>D3</b>	3.542	0.962	0.097	4.422	0.783	0.179	18.6
<b>D4</b>	4.404	1.434	0.010	5.613	1.199	0.235	16.4
<b>D5</b>	3.539	1.981	0.063	5.262	1.660	0.321	16.2
<b>D6</b>	3.978	2.101	0.096	5.824	1.750	0.351	16.7
<b>D7</b>	3.344	1.158	0.016	4.350	0.990	0.168	14.5
<b>D8</b>	3.551	1.677	0.081	5.054	1.422	0.255	15.2
<b>D9</b>	3.277	2.998	0.012	5.855	2.566	0.432	14.4
<b>D10</b>	3.606	0.690	0.032	4.239	0.601	0.089	12.9
D11	3.106	2.959	0.086	5.760	2.568	0.391	13.2
<b>D12</b>	3.836	1.207	0.060	4.924	1.028	0.179	14.8
D13	4.052	1.210	0.043	5.134	1.039	0.171	14.1
<b>D14</b>	3.251	1.956	0.001	4.922	1.670	0.286	14.6
D15	3.245	0.880	0.051	4.054	0.758	0.122	13.9

# **Appendix 4: Large-scale Treatment**

Table A 11: Lamort pulper specification.

Part: Motor						
NL351365015/5		3 phase motor				
50 Hz	type MBT 132	SA - 4/8				
3.7/2.2 kW		- hp				
1400/700 rpm		C F				
400 VYY		7.5 A				
$400~\mathrm{V}\Delta$		7.2A				
Cos φ 92/0.62	IP 55	IEC 34:1				
Calent/Temp rise		Class B				
Part: 2-speed Variable Speed Drive						
Model	PDL Microdrive 3					
Part: Variable Speed Drive						
Model	Altivar-21					
U (V ~)	380/480 Ø3	380/480 Ø3				
F (Hz)	50/60	0.5/1000				
I (A)	66	66				
U (V ~)	460/480 Ø 3	460 Ø 3				
F (Hz)	50	0.5/1000				
I (A)	56	FLA 52				
Short circuit withstand 5000 A, 480 V when fitted with fuse						

Table A 12: Centrifugal pump technical information.

Variable	Value	Notes		
Pump	Scanpump	Made in Sweden		
Bearing	3	Bearing assembly size		
$\mathbf{D_s}$	0.10 m	Suction size		
$\mathbf{D_d}$	0.11 m	Discharge size		
$\mathbf{D_i}$	0.37 m	Impeller size		
Phase	3 ~	_		
$\mathbf{F}$	50/60 Hz	Frequency		
P	22/25 kW	Power		
N	1475/1775 rpm	Rotational speed		
Cos $\varphi$	0.81	Power Factor		
Q	$250 \text{ m}^3 \text{ h}^{-1}$	4167 LPM		
H	35 m			
N	1470 rpm			
VY	380 - 420/440 - 480  VY	44/44 A		
${f V}\Delta$	$220 - 240/250 - 280 \text{ V} \Delta$	76/76 A		
Control	Altivar 61 variable speed control	ATV61HD36N4S337		
P	30 kW	Motor power		
Dimension	240 x 550 x 266 mm	Size for model IP20 S7A		

Table A 13: Comminution enthalpy calculations.

	Time (h)	Temperature (°C)	Enthalpy h <sub>f</sub> (kJ kg <sup>-1</sup> )	ΔΤ	Q (kW)	Δh (kJ kg <sup>-1</sup> )
Experiment	0	14.7	61.67			
1	1	37.1	155.39	22.4	2600.89	93.71
	2	45.5	190.53	8.4	975.33	35.14
	3	54.4	227.76	8.9	1033.39	37.23
Experiment	0	13.3	55.82			
2	1	42.5	177.98	29.2	3390.44	122.16
	2	54.4	227.76	11.9	1381.72	49.79
	3	63.4	265.42	9	1045.00	37.65
	4	67.6	282.99	4.2	487.67	17.57

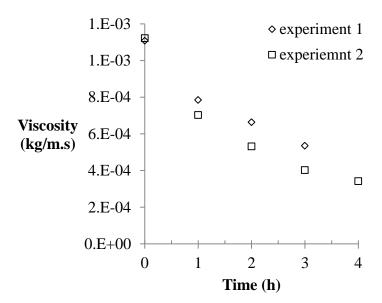


Figure A 5: Bulk slurry viscosity during comminution.

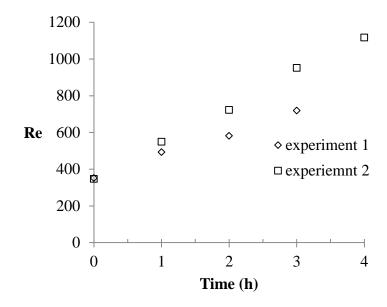


Figure A 6: Slurry Reynolds number during comminution.

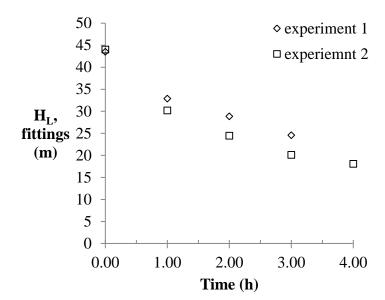


Figure A 7: Comminution system head loss due to fittings.

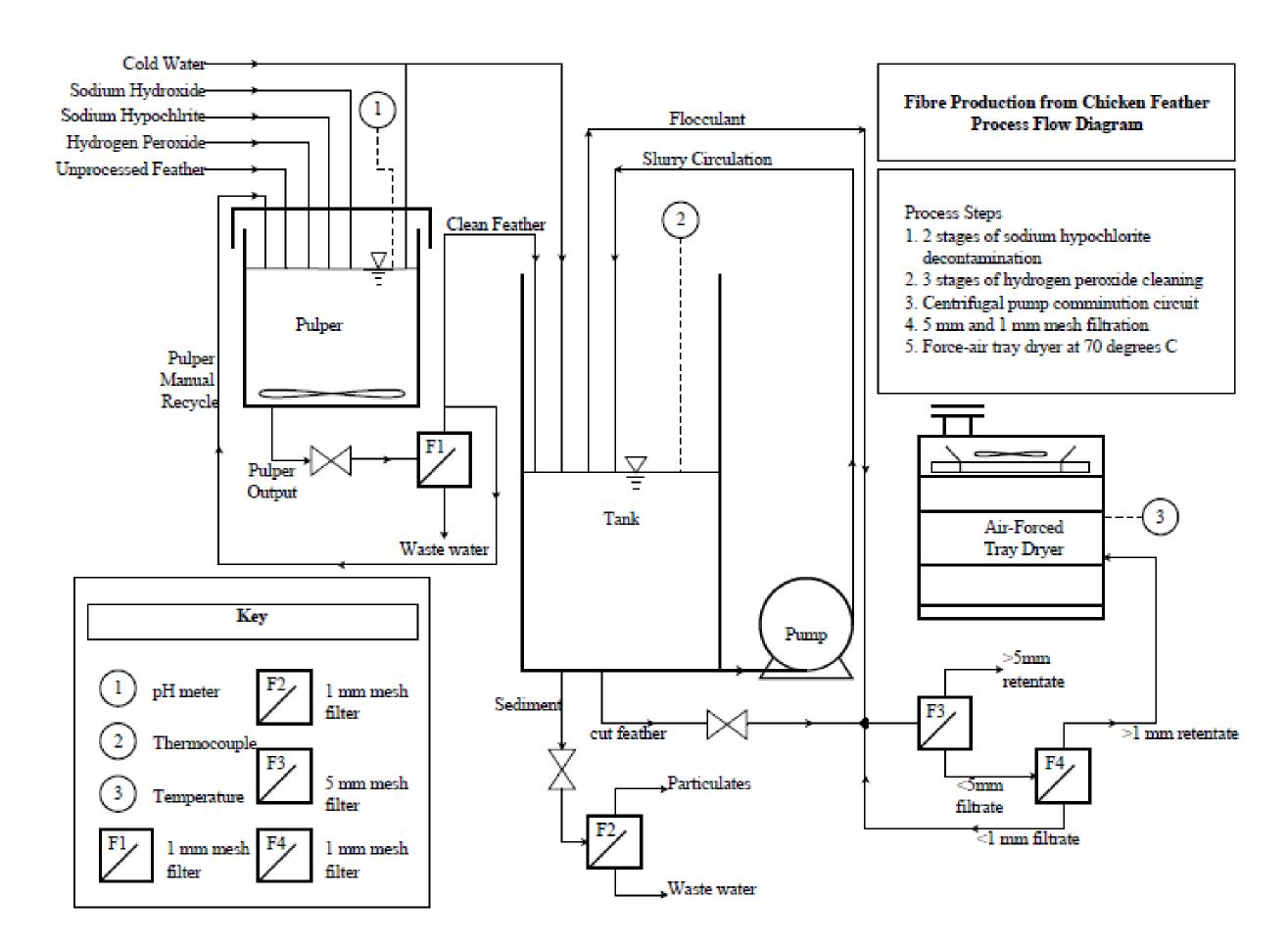


Figure A 8: Process flow diagram.