THE VALIDITY OF PHYSICAL AND CHEMICAL TESTS FOR DETERMINING THE QUALITY OF FLAX FIBRE PRODUCED IN VARIOUS TYPES OF RETTING

A Thesis

by

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PREFACE

As the work for this thesis was done at the Pilot Flax Fibre Mill, Portage la Prairie, Manitoba, a short history and the purpose of the mill will be given. The Dominion Minister of Agriculture, the Honourable J. S. Gardiner, strongly supported by other members of Parliament, felt that the chemurgic problems of the prairie should be thoroughly investigated. As a result of their endeavours, Parliament voted a sum of money for this purpose. Among these chemurgic problems, the utilization of flax straw was considered one of the most important.

In the spring of 1944 the building of a Pilot Flax Fibre Mill was authorized under the Experimental Farm Service. This mill was to function as a research centre for the study of all classes of fibre, but mainly flax fibre and its by-products. The Chief of the Fibre Division of the Dominion Experimental Farm, Mr. R. J. Hutchinson, made a survey of Manitoba and Saskatchewan to determine the location best suited to this project. Portage La Prairie was selected because it had been shown that fibre of good quality could be produced in this area.

A modern 1000-acre unit mill was erected and completely equipped with the latest in flax processing and seed cleaning machinery. In addition, two of the latest type water-retting tanks of 15,000 gallon capacity were built, and seeding and harvesting equipment was purchased. The project includes a laboratory equipped to carry on research in both chemistry and bacteriology. Dr. J. C. Woodward of Dominion Experimental Farm Service was appointed chemist in charge. It was due to the co-operation of Mr. Hutchinson and of Dr. Woodward that I was able to work at the Pilot Flax Fibre Mill and thereby gain a greater insight into flax fibre problems.

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Introduction

Grading of flax fibre in Canada, and in all other flax-producing countries, is done by professional graders who judge the flax by appearance (cleanness, glossiness, length of fibre, colour), by handle or feel (weight, softness), and by strength (breaking a few strands of fibre in the hands). Because this method is based on human judgment, two graders may award different grades to samples of the same fibre. It was felt that if some scientific method for ascertaining grades could be obtained, it would be a valuable contribution. Hence, a series of chemical determinations was made on flax fibre of various grades produced by water and dew retting in an attempt to assess the quality of flax fibre by chemical analysis. Concurrently, use was made of the Pressley Cotton Fibre Strength Tester to determine whether or not this machine could be adapted to flax fibre and yarn to differentiate between grades by means of physical tests.

An investigation was made also of various types of chemical retting procedures. Because of the speed with which flax straw can be retted by chemical solutions, an attempt was made to determine if fibre of quality equal to that obtained by water and dew retting could be produced thereby. The chemically retted fibre then was subjected to the physical and chemical tests used in the attempts to appraise flax produced by water and dew retting.

It was the purpose of the study presented in this thesis then, to attempt to correlate either or both a physical testing and a chemical testing with observed fibre quality. Such a test could serve to provide an easy routine determination of flax fibre quality in any laboratory, the conclusion being drawn from reproducible and unbiased results. Also, an attempt was made to investigate various chemical retting procedures on Canadian flax.

HISTORICAL

1. Retting

The Purpose of Retting

The stem of the flax plant consists mainly of a woody core and a surrounding cortex which contains the bast fibre bundles. Retting is said to be the process by which the pectic material which binds the fibres to the remainder of the stem is broken down, and the fibres are liberated. When flax is immersed in water, the straw becomes softened and the soluble constituents, which include carbohydrates, glucosides and nitrogen compounds, are extracted. These provide for abundant growth of the micro-organisms naturally present on the flax plants. The retting is brought about by certain types of bacteria which break down the pectic complex of the middle lamella of the flax plant. The fibre bundles are first separated from each other and from the cortex and then are themselves split lengthwise into fibres. The fibres vary in length from 11.2 to 38.4 inches and these are composed of flax cells or ultimate fibres from 1.0 to 1.2 inches in length. If the flax is under-retted, the fibres will adhere to each other in bundles giving a coarse, harsh fibre. Also bits of bark or shive may be found clinging to the fibre too tenaciously to be removed in the next operation which is scutching. If over-retted, the fibres will be attacked with partial breakdown into cells or ultimate fibres, resulting in the production of short, weak fibre.

Methods of Retting

Retting can be achieved by various methods: a) dew retting, b) pond retting, c) river or stream retting and d) tank or water retting (with or without the addition of bacterial cultures) and e) chemical retting. Each of these methods will be discussed.

Dew Retting: The pulled flax straw is spaced evenly on the ground and must be turned regularly by hand or by machine. Atmospheric

conditions are most important and those which are most favorable include a suitable supply of moisture in the form of dew and dampness from the grass and soil below, warm nights and not extremely hot days. The retting is allowed to proceed for several weeks, the time being governed by atmospheric conditions. The fibre produced is of a grey colour. This is the cheapest form of retting and is usually carried out by the individual farmer.

It is believed that the organism responsible for dew retting is largely aerobic in nature. The work of Behrens (13), reported in 1903, has formed one basis for subsequent investigations. His findings have been confirmed by Ruschmann (62) who stated that Mucor plumbeus and Cladosporium herbarum are the active agents with the latter being more widely distributed and more active. Also, Muller (47) included the giant mould Mucor stolonifer genannt and Behren's Mucor hiemalis which rets at a low temperature of 20° - 50° C, the optimum being less than 30° C. Jensen (35) reported that the fungi Dematium pullulans, Cladosporium herbarum and Alternaria sup. were the true agents of the dew-retting process in Australia. In summing up dew retting, Ruschmann (62) concluded that the uncertain issue of the ret, the dependence upon weather, the fact that a good quality of fibre is seldom obtained, and that a diminished yield of fibre is produced, are all against using this method of retting with the best quality of flax straw. The cost of dew retting is less than with warm water tanks as is borne out by Tobler who concluded that from 50 - 100% more labour is required in the factory retting of flax by the common European processes than is necessary in dew retting as practised by farmers.

Several patents have been issued for processes which undertake dew-retting under artificially controlled conditions. One of these (German patent #8-P.340,418) developed by Ochmann, consisted of packing the straw in tanks and spraying it with water for two hours every other day. The same water was used repeatedly, being subjected to a simple oxidizing treatment. The removal of a large proportion of impurities was brought about by allowing them to settle out between sprinklings.

Cromer's process (U.S. patent #1,448,391, March 13, 1923) required stacking the flax in superimposed layers in an inclined position when harvested. After curing, moisture was added to facilitate retting, after which the stack was aerated.

A third process, that of De Geyter (Belgian patent #364,851, Nov. 30, 1929 employs a combination of water retting followed by dew retting. The flax straw is immersed in a solution consisting of diluted liquor in which flax has been macerated and to which has been added nutritive substances and sporulated cultures of active Mucor. The material is warmed for 1 - 2 days and then spread out in the open or in rooms at 30° C. in which the air is kept moist.

None of these processes is in use today. The extra handling and equipment added to the cost of production without any improvement in quality have made these methods obsolete.

Pond or dam retting: In Ireland and in some parts of the continent, flax is steeped or retted in earthern dams or ponds of any convenient length and about nine feet wide and four feet deep. The bundles of flax straw are packed in almost vertically with the top end up. Mud or sods are then placed on top and the whole kept down by stone, so that the beets or bundles are covered by about three inches of water.

In the Lokeren and other "blue" districts of Belgium, as well as in Holland, the mud which accumulates at the pond year after year is employed to cover the top layer of flax. When the filling of the dam is completed, the top surface presents a level expanse of soft black mud. The main purposes of the mud are: 1) to keep off the light and thus prevent discoloration, 2) to give the fibre the requisite blue or black colours, 3) to make the retting process take place more uniformly throughout the entire mass, 4) to serve in lieu of stones to keep the flax straw under water. Upon completion of the ret, each beet is given one or two plunges in the water to remove soil. In this type of retting, the water is stagnant throughout the ret, with no addition of water.

River or stream retting: Flax retting proceeds quite successfully in a running stream of fresh water. The best flax fibre produced is retted in the River Lys in the Courtrai district in Belgium, while its substitute, "white Dutch" is retted in the River Scheldt in Holland. The success achieved is probably due in great measure to the slow movement of the water and the exceptional bacterial development resulting from continuous retting. Under the Courtrai system, the sheaves of dried straw are packed horizontally or vertically in large wooden crates lined wi th straw or sacking. The upright position is usually adopted as it is said to be more favourable to the production of light-coloured fibres as no sediment can rest upon it at any stage of fermentation. The loaded crates are then slid into the water, anchored and weighted with stones so that they are submerged a few inches below the surface. As a rule, after steeping for a few days, the flax is removed from the crates and set up in stooks to dry. The advantage of the interruption is that exposure to the sun and air kills the microbes of putrefaction which have developed. Hence the strength of the fibre remains unimpaired. When the straw is dry, it is packed in the crates and returned to the river. In seven to twelve days the retting is completed.

A variation of river retting is canal or channel retting, a system devised by Schneider. It consists of a continuous process in which fresh flax bundles pass slowly through a canal in a slow current of water so that by the time they reach the end they have been retted sufficiently. The Feuillette system, as described by Durant (28), Muller (47), Ruschmann (62) and others, employed the channel system of retting modified to the extent that the crates containing the bundles of flax were raised intermittently out of the retting water, thereby aerating the straw. This encouraged the development of aerobic organisms which were believed to be the real retters, while the activities of anaerobic organisms were discouraged. This system did not gain widespread use because of the fact that lots of straw which varied as to its stem diameter and maturity, could not be treated differently than the standard straw. Also, the process included a drying system which was prohibitive from an economic standpoint.

Recently Lüdtke (44) performed tests comparing flax retted in stagnant water to flax retted in flowing water. He found that the colour and fineness of the latter were slightly better, the feel was inferior, and the strength and yield were the same as in stagnant water.

Tank or warm water retting: The earliest information we have of tank retting was given in a report by a Mr. McAdam, Secretary of the Old Royal Belfast Flax Society in 1846, as recorded by Carter (17). This report described Schenk's flax rettery which was established on the Newport River, county Mayo, Ireland. Large vats four feet deep were used; these had false bottoms, underneath which lay steam pipes. The flax was packed into the empty vats and battened down. Water was let in to fill the tanks and a frame was fastened on the top to prevent the rising of the flax in the course of

fermentation. Steam was released from the pipes for eighteen to twenty hours until the temperature of the water reached 32°C. Retting was completed at the end of sixty hours.

Previous to this time the only heat in retting waters was obtained from the sun's rays. The intensity varied from day to day and with the seasons, hence the retting periods were frequently prolonged.

Eyre and Nodder (29) found retting to be most rapid at 37°C. Above 40°C. and below 20°C., the action of normal retting bacteria is inhibited and the rate of retting decreases. In general it has been found that between 25°C. and 32°C. the most satisfactory results are obtained. Higher or lower temperatures have been used to promote retting by specific organisms as in the Carbone process, using the organism Bacillus felsineus where a temperature of 37°C. was required. The danger of over-retting is much greater at higher temperatures.

The Belgian retting tanks, on which many retteries of the world are modelled, consist essentially of four large concrete tanks, well insulated by having their walls interlined with cork. Each tank has a capacity of about four tons of deseeded flax straw. Above the retting tanks are hot water tanks, and above these are cold water tanks; these provide for adequate control of the temperature of the retting water. This tank was developed following legal restrictions on the use of the River Lys. It has been shown that flax fibre obtained from the controlled rets in these tanks was equal in quality to that retted in the Lys itself, so it may be stated that tank-retted flax, properly controlled, represents the highest quality of flax known at present.

In order to control the end-point of retting and to avoid weakening

of the fibre through over-retting, various procedures have been introduced to retard bacterial growth in the later stages of retting. These are all based on the principle that withdrawal of leach liquor, or periodic replacement of part of the retting liquor with fresh water, removed a large proportion of the nutrient material upon which active bacterial growth depends.

There are a large number of tank-retting processes, wide differences of opinion as to the relative merits of each and constant attempts to introduce improvements.

In Ireland, Searle (65) in 1929 recommended a standard ret of six days at 22°C. with a flow of 1 gallon of water per ton of straw per minute. Turner (69), Director of Research of the Linen Industries Research Association, stated that retting and scutching plants on Belgian lines have been established in Northern Ireland and that the services of a Belgian expert have been engaged. He also mentions that a modified retting method has been devised recently, the "Interrupted Ret". It is claimed that this ret is easier to control than the ordinary Belgian ret, as it requires less supervision and is less critical at the end point. The fibre strands are more subdivided and the flax produced is of a slightly higher quality.

In Australia, much serious study has been made of flax problems. Most of this work has been done at the Flax Research Laboratory (formerly the Flax Section of the Division of Forest Products) operated by the Council of Scientific and Industrial Research. Munro (49) recommended a preliminary rinse for ten to seventeen hours with water at about 25°C. This was to be followed by retting with fresh water at 30°C. to 40°C. for six to seven days with dilutions of the water at intervals.

In the United States, in Oregon, tank retting has been carried out in the following manner. Sufficient water at 27°C. is added to the concrete tanks packed with flax straw. The flow is controlled so that the water in the tanks is completely changed every two days. At the end of four or five days retting is completed.

In Canada, at the Pilot Flax Fibre Mill, Portage La Prairie, Manitoba, much experimental study has been made on tank retting. It would seem that a satisfactory treatment consists of retting at 25°C. with sprayings to maintain the initial temperature and a final cold rinse to remove the organic acids from the straw, thereby greatly reducing the dust in later handling.

The tank rets described above have made use of fresh water in the initial filling of the tanks and for any additions. However, portions of old retting liquor have served as a source of inoculum for the water of new rets. Carter (18) described the retting procedure of Legrand Vansteenkiste at Wevelghem Belgium, in which use is made of old retting liquor in the first stage, later to be replaced by fresh water. Muller (47), mentions the Couisinne process which was used in France to a limited extent. This consisted essentially in adding a portion of old liquor to the new retting solution, as well as milk of lime to neutralize the acidity.

Another example of this type of retting was that of Lucas (British patent #339,808, Dec. 23, 1929) in which a quantity of liquor from a previous first stage ret was added to the new retting water at a temperature of 25°C. which was maintained for twelve to fourteen hours. A portion of the liquor was then withdrawn (to be used for the next batch), the tank filled with fresh water and the temperature slowly raised to 37°C. at which it was maintained until retting was completed.

In England, during World War II, some interesting experiments were carried out with regard to re-usage of retting water (3). The effluent from

anaerobic retting (all tank retting described above is of this type) is highly polluting and evil-smelling due to the presence of butyric acid. It was found that when the liquor was adequately aerated during retting by means of a current of diffused air, it could be re-used for retting, for at least forty successive batches, without discharge of waste waters. The liquor remaining was only two or three times as polluting as the liquor from a single anaerobic ret, was approximately neutral in reaction and comparatively inoffensive in character. Aeration of the contents of the retting tank encouraged the growth of aerobic and facultative organisms, but it was significant that the number of anaerobes still remained high unless the rate of aeration was excessive. Bacteria, capable of causing soft rot in potatoes, increased considerably during the course of the ret. Results of an investigation by Allen suggested that organisms with this characteristic were also responsible for the retting action, both types of change being due to the breakdown of the pectic complex of the middle lamella of the plant tissue. In both anaerobic and aerated rets, spore-bearing anaerobes appeared usually to be the active agents in retting, and the majority of strains isolated in pure culture from these rets (which were operated at 30 °C.) possessed the characteristics of Clostridium tertium. The count of spore-bearing aerobes was usually negligible, but under conditions of excessive aeration, B. subtilis appeared in large numbers.

Under anaerobic conditions, growth of micro-organisms resulted in the formation of considerable quantities of organic acids, neutral volatile compounds and gases. The accumulation of these metabolic products tended to inhibit further growth of bacteria and hence prevented the re-use of liquor of an anaerobic ret. The aeration of the liquor altered the metabolism of the bacteria so that more complete oxidation of the organic matter occurred, carbon dioxide and water being formed in place of organic acids and other metabolic products.

Retting with addition of pure cultures: The earliest record of this type of retting is given by Carter (19) who stated that Doumier in 1899 advocated a method of retting in warm water to which had been added bacteria isolated from retting liquor. Bazzochi (11) mentioned that Deumaer and Deswarte employed a culture of Bacillus amylobacter to ret flax with good results. Bazzochi also states that Hiltner and Stormer used cultures of Plectridium pectinovorum and other organisms of secondary value while Mackrinoff used Pectinobacter amylophilus in his process.

At the beginning of this century, Rossi (61) and his collaborators started extensive investigations at the Institute of Agricultural Bacteriology in the Royal Higher School of Agriculture at Portici, Italy. The retting process which he introduced consisted of the following steps:

1) Immersing the flax straw in ordinary water.
2) Raising the temperature to 28°C. to 30°C.
3) Adding a suitable quantity of Bacillus comesii (aerobic)
4) Passing a current of air through the retting water for the duration of the process by means of perforated pipes at the bottom of the vats.

The main advantages were the shorter period necessary to complete the retting process and the eliminating of the danger of over-retting. Ruschmann (62) found that a very intense aeration, many times that specified by Rossi, was necessary, which entailed expenditure not justified by the product. The fibre was found to be duller in appearance, harder, darker in colour than that obtained in the anaerobic ret.

Another Italian, Carbone (16), isolated from the mud of retting pits an anaerobic bacillus he called Bacillus felsineus. This he used to inoculate the retting water which was maintained at 37°C. Tobler (68) evaluated the procedure as follows:

Advantages

1) Reduced the time required to complete the retting process.
2) Improved the colour of the fibre.
3) Reduced odour and acidity of retting liquor.

Disadvantages

1) Additional labour required to prepare inoculating material and to inoculate retting water.
2) Extra cost of maintaining high temperature.
3) The danger of over-retting.

As late as 1939, it was stated (15) that the Italian fibre industry was using Bacillus felsineus Carbone (Clostridium felsineum) almost exclusively for the steeping of hemp and flax. Ruschmann and Bartram (64) on experimenting with the addition of B. felsineus to the retting water found that resistant flax, ordinarily requiring seven to nine days, was retted in five days, producing cleaner and brighter fibres.

Gibson and Whiting (30), when recently investigating the use of hiparol [earlier discovered by Baruah (10)], found this enzyme complex consisting of lamellase, proto-pectinase and pectinases in a 3.6% suspension produced considerable acceleration of the retting process.

Retting with the addition of pure cultures has not been used to any high degree. It has been found generally that the organisms responsible for retting, and present naturally in the flax straw, increase almost as strongly without inoculation as with inoculation. The main advantage, acceleration of the process, has not warranted extensive use of this type of retting.

Chemical retting: Much time and effort have been spent in attempts to achieve retting by chemical processes. Bazzochi (11) mentioned those of Professor Pietre Willermoz di Liene and Abbott Rezier in 1786 in which organic acids and sulphur were the active reagents. In 1789, an unknown member of the Patriotic Society of Milan claimed that hemp could be retted with vinegar and the yolk of an egg. Further, Bazzochi reported that Thummler, Leidel and Baur used alkalies acids, while Nicelle and Schmidt of London, de la Roche of Paris and Sampson proposed methods based on the use of acid salts of soda and soap.

The Cheveline Process (4), which consisted of treating the flax with alcohol followed by mineral oil and steam, was tried near Moscow, but was

unsuccessful. Another process which did not stand the test of time, the Rosseau process (5), comprised a bleaching treatment followed by maceration in a solution composed of sodium carbonate, sodium sulphate and soap containing petroleum ether. The Rogers process (6), which proved to be unsatisfactory because the fibre produced was weak and uneven, required an hour's boil in an emulsion of linseed oil. Bradshaw (14) developed a process which consisted of alternate alkali and acid treatments, the carbon dioxide generated splitting the fibre into ribbon-like filaments. An unnamed process (7) used milk casein and sodium carbonate to ret flax and it was claimed that greater strength was retained and improved dyeing qualities acquired thereby.

Hydrocarbons have been used as retting agents to some extent. The Douglas process (U.S. patent #1,224,722, 1917) was based on the use of kerosene and gasoline. The fibre produced was coarse and harsh. The Peufaillit process consisted of using about 4% petroleum oils under pressure in autoclaves. An extensive description of it was given by Ruschmann (63) who criticized the process by stating that equally good results were obtained by the use of steam alone.

Kidger and Harris (British patents #190,198, May 15, 1922) used soap to degum various textile fibres. A small quantity of paraffin was admitted at the bottom of the tank to cause the separated resinous and vegetable matter to pass away through an overflow outlet. Later a small quantity of benzine or gasoline was added and boiling was continued until digestion was completed. This process has not been used to any extent.

Lowry (43) experimented with soap retting but found the soap difficult to remove and experienced some trouble in scutching due to a matting of the fibres in drying.

Johnston (36) found in his work that although a creamy white fibre of fairly good quality was produced when soap was used to ret flax, the adhering soap materials were very difficult to remove. Washing with warm water gave better

results than with cold water, dilute acid, alkali or ammonium oxalate solutions. However the amount of warm water required to remove most of the soapy materials was prohibitive from an economic standpoint.

Munro (50) has reported encouraging results from chemical retting carried on in Australia. One treatment consisted of a six hour boil in N/50 HCL followed by a six hour boil in N/50 NaOH to which has been added 0.125% of oleic acid. Munro explained the action as hydrolysis of certain cementing substances, solution of some materials, and, through the emulsifying action of the sodium oleate, suspension and ultimate removal of certain constituents. The oleic acid or sodium oleate probably replaced the natural waxes removed during the reactions and gave the necessary oiliness to the finished fibre. He found that the action of oleic acid was specific and that no other oils or fatty acids have been discovered which will replace it. Although this process has reduced the retting time to twelve hours, the cost of treatment (£2 per ton of straw) remained as a disadvantage.

Primot (56) recommended treatment with boiling 0.5% - 2% solutions of sodium, potassium or ammonium citrate, oxalate, fluoride, arsenate or phosphate as a convenient method for degumming flax or ramie. He assumed that soluble pectates and insoluble calcium salts were formed, thereby loosening the fibres.

Recently, Taylor (68) of the Georgia School of Technology has carried out various experiments in the retting of flax straw. The treatments investigated included steaming, cooking in solutions of 0.5% and 1% sodium sulfide, sodium carbonate, sodium hydroxide and acetic acid. In addition, mixtures of sodium hydroxide and sodium sulfide, sodium carbonate and sodium sulfide were tried. None of the resulting fibres compared favorably with water-retted fibre.

It would seem that several more years of research will have to be spent before chemical retting could replace water retting to produce the best fibre.

II CHEMICAL ANALYSES

Over a period of thirty years a vast literature has accumulated on methods for determination of cellulose and lignin in fibrous materials. The greater part of this literature is reviewed in "Wood Chemistry" edited by Louis E. Wise and published by Reinhold Publishing Corporation in 1944. It is not proposed here to enter upon a thorough review of this literature but rather to select and present briefly the essentials of the procedures which have been considered the most desirable.

Cellulose Determination

Essentially it can be seen that an acceptable determination of cellulose is contingent upon a successful removal of the lignin with which it is closely linked in the plant cell wall. Therefore, the methods which are presented here for cellulose analyses are procedures to achieve the separation as completely as possible without entailing any loss of cellulose. The converse will hold as will be seen for lignin determinations.

For cellulose determination, the following procedures have been recommended: In the Cross and Bevan method (25), 5 grams of powdered material are heated for 30 minutes at the boiling point with 100 ml. of a 1% sodium hydroxide solution, the volume being kept constant with the addition of water. After filtering and washing, the residue is spread out in a beaker into which a slow stream of water-washed chlorine gas is passed. At the end of 30 minutes, the residue is filtered and washed to remove hydrochloric acid and then boiled with a 2% sodium sulphite solution to which 1% sodium hydroxide is added. After filtering and washing, the last traces of lignin are removed from the cellulose with 0.1% potassium permanganate or 0.1% hypochlorite solution. The isolated cellulose is given final washings with a sulfurous acid solution and with water.

In the Kurschner and Hoffer method (39), the sample is given repeated

treatments with a 20% solution of concentrated nitric acid in 95% ethanol until purified cellulose is obtained. The end point is determined by treating the residue with a phloroglucinol hydrochloride solution which, in the presence of lignin, gives a cherry-red colour. The extraction with the alcohol-nitric acid mixture is repeated until a negative colour reaction is obtained. As a modification, Kürschner and Hanak substituted acetic acid for the alcohol in the digestion reagent in order to reduce the time of treatment.

Norman and Jenkins (51) extracted the sample with an alcohol-benzene mixture, then immersed it in 100 ml. of 3% neutral sodium sulfite solution which is heated to boiling. After filtering, the sample is removed to a beaker, 100 ml. of water are added followed by 5 ml. of sodium hypochlorite (15% available chlorine) and the suspension is allowed to stand for ten minutes. The residue is then separated, transferred to a beaker, and made up to 50 ml. with distilled water. Fifty ml. of 6% sodium sulfite are added and the mixture boiled for 20 minutes. After filtering and washing, the neutral hypochlorite treatment is repeated followed by another sulfite treatment. The material is now suspended in 100 ml. of water and 5 ml. of dilute sodium hypochlorite solution (3% available chlorine) are added together with 2 ml. of 20% sulfuric acid. Chlorine gas is evolved and the material is subjected to a 10 minute treatment in the solution before it is filtered and washed. The residue is made up to 50 ml. with water and treated with sodium sulfite as before. An intense purple colour is obtained indicating the presence of lignin. The acid hypochlorite treatments are continued as long as positive reaction for lignin is obtained. After a final hot water wash, the product is dried to constant weight.

Finally there is the monoethanolamine method of Wise, Peterson and Harlow (74). Here a 1 gram sample is treated with 50 ml. of monoethanolamine for 5 hours at 170°C., followed by bleaching with chlorine water and digesting

with hot sodium sulfite for 30 minutes. The residue of cellulose is washed and dried to a constant weight. In this connection, Reid, Nelson, and Aronovsky (58) have shown that the use of hot monoethanolamine yields satisfactory results for farm wastes. It is believed that this technique should be investigated further because results have indicated that the action of monoethanolamine is considerably gentler than that of chlorination.

In the course of an attempt to arrive at the best procedure for cellulose determinations, Reid and Lynch (57) conducted a critical comparison of the Cross and Bevan, the Norman and Jenkins, and the Kürschner and Hoffer methods for the determination of cellulose in a variety of fibrous materials. Because of the short time required, the alcohol-nitric method is recommended by these authors for large numbers of routine cellulose analyses, particularly in agricultural wastes when no further determinations on the resultant cellulose is contemplated.

Crampton and Maynard (24) have applied the Kürschner and Hanak (38) method to routine cellulose analyses. They found by using alcohol instead of water for the first washings to free the cellulose from the digesting reagent that the process could be carried out by centrifuging and this considerably facilitated the washing operation. It was this adaptation of the Kürschner and Hanak method which was used in these studies for cellulose determinations in flax fibre.

Lignin Determination

From a consideration of the intimate association which lignin has with cellulose, hemicellulose and other polymeric carbohydrates of the plant cell walls, and also of the complex lipide and nitrogenous constituents of the dead cell remaining clinging to the cell wall, it is at once obvious that the absolute determination of lignin is fraught with

considerable difficulty and at best can give results of only approximate value.

In the first place when a lignin determination is desired, it is necessary to free the sample from fats, waxes and complex nitrogenous materials like protein which as indicated are probably the remains of the dead cells in and around the fibres. The removal of the fats and waxes does not present great difficulties. The solvent generally used is 1:2 benzene-alcohol (Phillips)(53). Extraction with ether has been recommended by Klason (37) and by Popov (54). Crampton and Maynard (24) first used ether and then, previous to the actual lignin determination, washed the material with alcohol and benzene. These solvents were used in the preparation of the flax fibre for lignin determinations as reported in this thesis.

The removal of the nitrogenous constituents presents greater difficulty. The methods must be drastic enough to dissolve proteins and similar material without appreciably effecting the solution of lignin. The following methods have been recommended:

(1) Williams and Olmsted (71) used pancreatin digestion as a preliminary treatment.

(2) Horwitt, Cowgill and Mendel (34) digested with pepsin, diastase and trypsin.

(3) Crampton and Maynard (24) employed pepsin digestion.

(4) Davis and Miller (26) digested the sample successively with pepsin, clarase and trypsin and then boiled it for 1 hour with 5% sulfuric acid before subjecting it to hydrolysis with 72% sulfuric acid.

The pepsin digestion method of Crampton and Maynard was adopted for use in the experimental work of this thesis. It is not suggested that this procedure offers an absolute method for removal of nitrogenous material.

In a complete and final analysis it would be desirable to present data both for what appears to be lignin and also for nitrogen in the lignin residue. For the purpose of this investigation, it was considered sufficient to determine lignin by the proteolytic method of Crampton and Maynard because it was believed that this method best limits the error introduced by the presence of extraneous nitrogenous substances. Therefore, the results are given for lignin alone.

In the second place, as suggested above, lignin as a distinct chemical identity has not been completely separated from cellulose and hemicellulose by the best of chemical procedures because it is so closely bound to cellulose in the cell wall and unlike cellulose does not occur independently in the plant. However, because of the great difference in the chemical properties of cellulose and lignin, an approximate separation and determination of the two can be achieved.

An analysis of lignin in plant material rests upon the successful application of those solvents which use to best advantage the differential solubility of cellulose and lignin. Therefore, in any attempt to estimate lignin in fibre consisting chiefly of cellulose and lignin, it is necessary either to dissolve the lignin leaving the cellulose as residue or to dissolve the cellulose leaving the lignin as residue. It would appear desirable to select the latter procedure for which the following methods are recommended in the current literature.

Klason (37) used 72% sulfuric acid to dissolve the cellulose for quantitative lignin determinations. In slightly modified form, this method has been used by Becker (12), Dore (27), Mahood and Cable (46), Paloheimo (52), Venkateswaren (70), Müller and Herrmann (48) and others.

Willstätter and Zechmeister (73) removed cellulose with fuming hydrochloric acid. This was applied later by Krull (37) to determination of lignin. Many modifications of this method have been reported. Goss and Phillips (31)

used fuming hydrochloric acid and dry hydrogen chloride gas in a special apparatus. In the method of Popov (54), the sample is first refluxed with 2.5% hydrochloric acid and then mixed with a reagent prepared by dissolving 40 gr. of zinc chloride in 100 ml. of 37% hydrochloric acid to which have been added 5 to 10 ml. of water. It has been claimed that this latter method gives results comparable with those obtained by the fuming hydrochloric acid method.

The acid solvent acting on the sample finally produces a viscous mixture of lignin and dissolved cellulose from which the lignin is usually separated by filtration. When the recommended 72% sulfuric acid method is used , the resulting mixture does not lend itself easily to filtration. The use of a granulating agent (chloroform-acetic acid) in the precipitation of the lignin to lessen the time necessary for filtration was proposed by Ross and Potter (60) and has been recommended by Baillie (9) in a microtechnique.

The presence of mineral acids in prolonged contact with dissolved free sugars, present as such in the material, or as a product of the acid hydrolysis of the cellulose, results in the production of an insoluble humin-like residue which might separate out with the lignin. Ross and Hill (59) recommended the addition of formaldehyde solution to the 72% sulfuric acid - sample mixture to reduce the time required for the hydrolysis of the carbohydrates so that the humin production is avoided.

The method finally adopted in the course of experimental work given, based on appraisal of all the methods, was that of Crampton and Maynard (24) which is presented in the experimental section.

Oils, Waxes and Gums

High quality fibre flax, according to the present methods of grading, has a definite sheen or glossy appearance as compared to the lower grades, which feel dry and harsh. It seemed probable that these differences in appearance and handling quality might be quantitatively assessed by fractional determination of oils, waxes and gums. Also, this information might be of use in assaying the effect of various chemical retting techniques on the quality of fibre finally obtained.

Various solvents for extraction of oils, waxes and gums have been used. Clifford, Higginbotham and Fargher (20) found that in 30 hours or longer hot extraction removed more waxy substances than could be extracted in the cold, and that, of the solvents used, chloroform, benzene and carbon tetrachloride in the order named were decreasingly efficient. Maclean and Maclean (45) pointed to the fact that alcohol often removes, with ease, fats and other lipids that are removed only in part or not at all by the usual fat solvents. Hess (33) stated that, in general, the maximum amounts of resins, fats and waxy substances are extracted from cellulosic materials with acetone or hot alcohol. He credited Schwalbe with the statement that equal parts benzene and alcohol are appropriate for the quantitative determination of resin, fats and waxes in cellulosic materials. Conrad (22) developed a new technique for determination of wax in cotton fibre. In this two-step process, the wax is first extracted with hot 95% ethyl alcohol and then transferred to chloroform through a phase-separation process in order to eliminate sugars, mineral constituents and other non-waxy constituents removed at the same time by the alcohol. The wax is extracted more rapidly by hot alcohol than by chloroform, the most rapid and adequate of a considerable number of wax solvents previously studied. The American Oil Chemists Society official method (1) for determining oil in a variety of materials requires a 5 gr. ground sample in a folded filter paper to be extracted with petroleum ether for three hours in a Butt extractor. For

crude fat determinations, the official method of the Association of Official Agricultural Chemists (8) comprises extraction of a 2 gr. sample with anhydrous ether for 16 hours. Couchman (23), in her investigation of the losses of flax straw during water retting, determined the solubles in sulfuric ether, benzene-alcohol (2:1) and alcohol. The Linen Industries Research Association (L.I.R.A.) (42) devised a system of analyses in which chloroform was used to extract fat and wax, and alcohol to extract gums.

In these studies, it was decided to extract the same sample with the following solvents in the order named:

(1) Petroleum ether (Skellysolve F.)
(2) Chloroform
(3) Ethyl alcohol (95%)

III Tensile Strength

The determination of the strength of a textile material gives very useful information. However, strength as an imitative test is of no value for in actual use a textile material is not subject to a slowly and steadily increasing load as is used in the strength test, but rather to abrasive action and smaller repeated loads. Strength as a measure of uniformity is a very useful test since a change in any physical property or a change in the chemical constitution of a textile material will nearly always result in a change in strength. It may be stated then that the principal value of the strength test is its use to measure the quality of the textile material, which is dependent upon the nature of the constituents of the material and upon the quality of the refining and processing.

Standard procedures [American Society for Testing Materials Standards (2), and Codes and Specifications, N.R.C., Ottawa (21)] are available for determining the tensile strength of yarns. These employ a machine of the pendulum type or of the inclined-plane type. Single yarn test specimens are used and jaws, clamps or capstans are set 10 0.25 inches apart at the beginning of each test.

Fibres may be tested either singly or in bundles. For testing the strength of single fibres, several testers have been offered but none of these has been accepted as an apparatus for standard procedures. The O'Neill single fibre tester consists essentially of a pair of clamps for the fibre of which the top one is fixed and the lower attached to a floating "bob". With the fibre in place, water is run out of the cylinder in which the bob is floating until the fibre is straightened; then a graduate is put in place. Water is run out of the cylinder into the graduate until the fibre breaks, and the amount of water indicates the breaking strength of the fibre. The Bowman and the Matthews fibre testers use an analytical balance in which one pan is replaced by the jaws while loading is done by placing weights on the other pan or by using a chain for weighting the other arm of the balance. Barratt's apparatus is also an equal-arm balance in which the loading is done by an electromagnet. Krais' apparatus is an equal-arm balance in which the loading is done by running water into a container on one side of the balance. Flax fibre does not lend itself to single fibre tests due to the extreme difficulty in separating a single fibre from the natural groups or bundles.

Bundle tests may be carried out by the Walen method, Grimes method, Chandler method or the Pressley method. The Walen method consists of parallelizing the fibres by pulling as for staple, brushing out all fibres less than 7/8 inch long, cutting the bundles to weigh about 0.004 grams. The ends of the bundles are cemented together with collodion and the bundles are broken in a 10 pound tester with $\frac{1}{2}$" gauge length. Five or more bundles are tested and the results are calculated to strength in ounces of fibre equal to a unit of #20 yarn. The Grimes method is similar to the Walen in that the fibres are combed out and cut to a definite length. The bundles are then adjusted to a weight of 0.025 0.005 gr., the ends are taped together with drafting tape and the bundles are broken in an ordinary fabric testing machine of pendulum type. The results are calculated to pounds strength per gram of fibres. In the Chandler bundle method, a bundle of fibres is combed out parallel and the bundle is wrapped with

#20 sewing thread on a special apparatus; the length of the thread for 10 revolutions is measured and the circumference of the bundle is calculated and corrected for the thickness of the thread. The samples are broken in a 300 pound capacity machine with special jaws and each break is corrected to a standard circumference of 0.125 inches. Each corrected break is then calculated to pounds per square inch. This method is the A.S.T.M. standard method for testing the tensile strength of cotton fibres. The Walen, Grimes and Chandler methods developed for cotton fibres are not suitable for flax fibres because they offer opportunity for slippage of the ultimate flax fibres so that a separation might occur between fibres rather than across them. The Pressley Cotton Fibre Strength Tester (55) offers a method of determining quickly and accurately the relative strength of cotton fibres. This very simple instrument is becoming widely used in the cotton industry. The clamps hold a standard length of sample 0.464 inches long and there is a straight cross-fibre break. The number of pounds read from the beam divided by the weight of broken fibres in milligrams gives the number of pounds required to break a milligram of fibre of a standard length of 0.464 inches. Williams and Painter (72) have made a study of variations due to day, operator and jaw fatigues in using the Pressley tester with cotton. Spencer (67) of the Howard Smith Paper Mills, Ltd., Beauharnois, Quebec, is using the tester in evaluating the strength of flax as well as of other fibres as a preliminary for processing for paper. There is a great need for a simple method of assessing the relative strengths of flax fibres because at present the grader estimates the strength by selecting a group of fibres and breaking it in his hands. It was considered that any simple quick method would have to measure the cross-sectional strength of a group of fibres of a known weight. Such a measure might not be related to the grader's estimations because the ultimate flax fibre is only 1 - $1\frac{1}{2}$" long and a group of these ultimate fibres are cemented together to form the long fibre as handled by the grader. It appeared that the Pressley Cotton Strength Tester might be

adapted for use with flax fibre to offer a quick, accurate method for determining tensile strength and for this purpose it was used in these studies.

Experimental

Since it was the object of this thesis to investigate the possibilities of developing, one, a physical, and two, a chemical test of flax fibre of various grades and rets, it will be convenient at this point to present the discussion of materials and methods in three sections.

First, a list of the various types of flax fibre selected for testing is presented. These are representative samples chosen from various locations in Canada where they had been subjected to water or dew retting. Listed with these are fibres produced by three chemical rets which were performed as part of the project of this thesis. These fibres were chosen from the most promising of a variety of chemical rets experimented upon.

Secondly, a discussion is given of the methods of chemical investigations of this fibre of different grades produced in the various retting processes.

Finally, the methods are described of measuring the tensile strength of the fibre produced in the same retting processes.

Materials:

(1) British Columbia water-retted fibre, grades C1 to C4 (decreasing in quality from C1 the highest grade) retted and graded by the Fraser Valley Fibre Flax Co-operative Association, White Rock, British Columbia (grades not official).

(2) Eastern Canada dew-retted fibre, grades C1 to C4, graded at the Economic Fibre Division, Central Experimental Farm, Ottawa.

(3) Portage La Prairie, water-retted fibre, grades C3 and C4, retted and graded by the Pilot Fibre Flax Mill, Portage La Prairie, Manitoba.

(4) Portage La Prairie, dew-retted fibre, grades C2 and C4, retted by the Pilot Fibre Flax Mill and the University of Manitoba, respectively, and graded by the Pilot Fibre Flax Mill.

(5) Fibre produced by three chemical rets:*

(a) Straw-retted in 0.5% ammonium oxalate.

(b) Straw green scutched first, and fibre retted in 0.5% ammonium oxalate.

(c) Straw boiled in 1.25% sodium hydroxide solution.

* (a), (b) and (c) are respectively rets #3, #9 and #15 in the table of chemical retting found below in Results.

Methods:

(1) Chemical determinations.

Preparation of samples: Representative samples of the whole fibre were ground in a Wiley Laboratory Mill, standard model, to pass a 1 mm. mesh sieve. Moisture determinations on each sample at the time of analysis showed that the fibre contained between four and five per cent moisture.

(a) Cellulose. [The Crampton and Maynard (24) adaptation of the Kürschner and Hanak (38) method]. A 1 gm. air dry sample was placed in a 150 cc. round-bottomed, wide-necked flask fitted with a reflux condenser. 15 cc. of 80% acetic acid and 1.5 cc. of concentrated nitric acid were added and the

mixture was boiled for 20 minutes. Sample and liquid were transferred to a 50 cc. centrifuge tube; 20 cc. of alcohol were added, and the mixture centrifuged for 10 minutes. The liquid was decanted and residue washed in the centrifuge tube with alcohol. The residue was transferred, with the aid of a stream of alcohol from a wash bottle, into an alundum crucible and washed successively with hot benzene, hot alcohol, and ether, using suction. The residue was dried, weighed, ashed and re-weighed. Cellulose was calculated as loss on ignition.

(b) Lignin (Method of Crampton and Maynard)

The oven-dry, ether-extracted residue from a 1 gm. sample was placed in a 50 cc. glass-stoppered Erlenmeyer flask and 40 cc. of a 2% solution of pepsin in 0.1 N hydrochloric acid were added. This was digested for 12 hours at 40°C., shaking frequently, especially during the first 4 or 5 hours. The non-digested residue was recovered by filtering through bolting silk and then washed successively with hot water, hot alcohol, hot benzene, hot alcohol and ether. The washed residue was transferred to a 100 cc. beaker and the last traces of ether were removed with mild heat. The residue was first moistened with 4 cc. of 40% formaldehyde and then 4 cc. of 72% (by weight) sulphuric acid were added and allowed to penetrate the sample for two minutes. 6 cc. of

concentrated sulphuric acid were added and the mixture stirred vigorously with a glass rod to aid in the solution of the sample which should be completed in 10 - 15 minutes. The beaker was partially immersed in a cold water bath to prevent the temperature from rising above approximately 70°C. When the mixture was dissolved, 35 cc. of a granulating agent consisting of a 1:6 mixture (by volume) of chloroform and acetic acid were stirred in, and then the whole was poured into 500 cc. of distilled water in an 800 cc. beaker. The contents of the beaker were boiled gently until the chloroform had been driven off (15 minutes) at which time the solution should have cleared and the lignin settled in granular form. This was filtered on a Gooch with suction and washed with not less than 200 cc. of 5% hydrochloric acid. The residue was dried and the lignin determined by loss on ignition.

(c) Ash [A.O.A.C. official method (8)].

A 2 gram sample was weighed into a porcelain crucible and placed in a muffler furnace previously heated to 650°C. This temperature was maintained for 2 hours with an automatic control pyrometer. Crucibles were then transferred directly to a desiccator, cooled and weighed.

(d) Ether, chloroform and alcohol solubles.

These were determined by extraction on a Goldfisch Extraction apparatus using the following solvents, in the order named, on the same sample

(a) Petroleum ether (Skellysolve F.)

(b) Chloroform

(c) Ethyl alcohol (95%)

The sample was extracted for four hours with each solvent. This extraction time was adopted since longer extractions did not result in any appreciable increase in amount of solubles. For example, the following material soluble in petroleum ether was obtained in the first four hours and in an additional four hours.

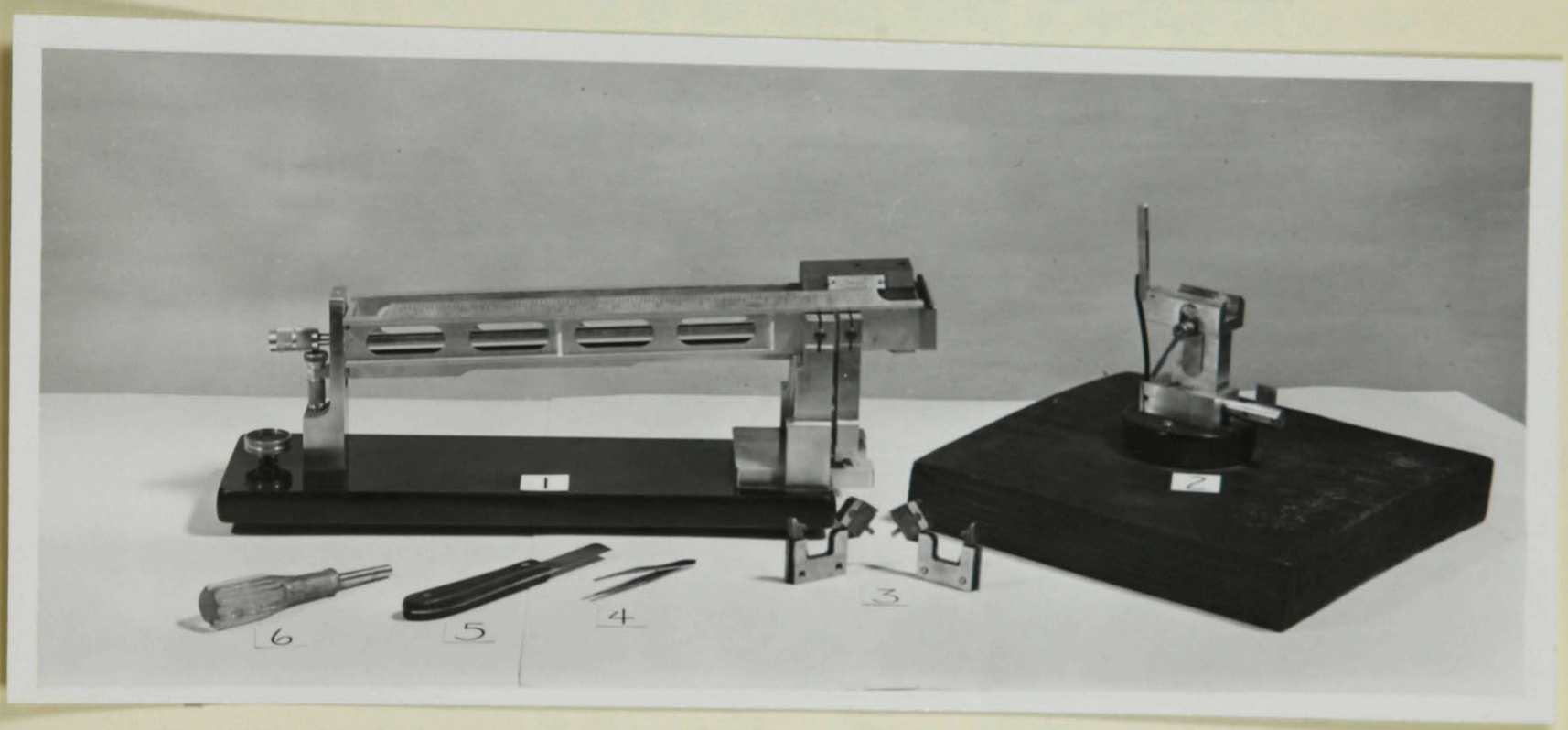
Sample number	1st 4 hours (%)	Additional 4 hours (%)
9	1.68	0.04
39	1.73	0.10
65	1.63	0.04
12	1.52	0.03

Samples were analysed in duplicate in all cases with duplicates on alternate days.

(2) Tensile Strength Measurement

A Pressley Cotton Fibre Tester (see Photograph) complete with two sets of clamps and a Christian Becker balance sensitive to 1/20 mg. were used for experiments in testing the tensile strength of flax fibre. A constant temperature and humidity chamber was not available, consequently samples of fibre were conditioned for 24 hours over a supersaturated solution of magnesium acetate before testing. Breaks (tensile strength determinations) were made in the laboratory at relative humidities varying from 40 to 60%. In carrying out a break, several strands of dressed flax fibre were selected at random from the hank to be tested, and combed slightly through the teeth which were mounted at the right side of the base of the vise (#2 in photograph). The halves

PRESSLEY COTTON FIBRE STRENGTH TESTER.



of the jaws (#3) were fitted together and placed in the vise. The mid-point of the strand of combed fibre was then placed on the lower leather holding-surfaces of the jaws, the upper sections of the jaws were lowered, and the screws evenly tightened with the wrench (#6). The pair of jaws, held together by the enclosed strands of fibre, was removed carefully from the vise, and placed upright on the wooden cutting surface (#2). Using the knife (#5), the fibre not enclosed by the jaws was cut off cleanly on both sides. This operation left an 0.464 inch length of fibre to be tested. While the beam of the Pressley machine (#1) was held in a horizontal position, the loaded jaws were inserted in the apparatus at the lower right end which projected over the base. The constant loading weight then was released; it travelled toward the left on the calibrated beam, and stopped automatically when the fibres were broken. The number of pounds required to break the fibre was read on the beam scale, the weight was returned to zero position, and the jaws were removed. The two halves of the broken fibre strand were removed from the opened jaws by means of forceps (#4) and weighed. The number of pounds required to break the fibre, divided by the weight of fibre in milligrams gave the "Index" of breaking strength ($\text{Index} = \frac{\text{Breaking strength in lbs.}}{\text{Weight of sample in mg.}}$)

RESULTS

I. Chemical Retting

The table below presents the originators, procedures, and the results of all the chemical rets experimented with. Rets were carried out both on the original straw and on green-scutched fibre. The latter was produced by passing unretted straw through a Forano Turbine Scutcher, thereby removing a large amount of the extraneous material. This would reduce the cost of chemicals used in retting, a factor which must be considered in any chemical processing. A rough evaluation of the quality of the retted fibre is represented by the letters A, B, C, and D in that order of quality. Successfully retted fibre is long, lustrous, soft and free from shive after dressing. Those rets marked with asterisks were chosen for chemical analysis because they were the most successful.

TABLE I

Ret No.	Originator	Retting Procedures	Results
1.	A.M.Munro	McCallister 1944 straw of Liral Dominion variety was boiled for six hours in 0.02N hydrochloric acid using a ratio of straw to retting solution of 1:15. Straw was then boiled in 0.02N Sodium hydroxide to which had been added 0.125% of raw linseed oil (calculated as % of the liquid volume) using a straw to solution ratio of 1:15. Linseed oil was substituted for oleic acid which was not available. Finally, the straw was boiled in water for one hour.	An appreciable amount of shive was still present after the sample had been passed through the line machine. Fibre was harsh*and of a medium grey-brown colour. C. *(linseed oil appeared to be ineffective in replacing natural oils)
2.	A.M.Munro	The same as above except green-scutched fibre was substituted for straw.	Same as above. C.
3.*	Howard T. Johnston	Maxwell's 1945 Liral Prince straw was treated with 0.5% ammonium oxalate solution on a steam bath for 4½ hours. Temperature was maintained at 80°C. Straw to solution ratio was 50 grams straw to 1 litre of solution. The loose core test was used to	No shive present but fibre was quite dry and harsh. 11% loss in weight after retting and air drying. B.

Ret No.	Originator	Retting Procedures	Results
		determine the end-point of the ret. Straw was then washed first in warm water and then in cold.	
4.	H.T.Johnston	The same straw as above was treated with a saturated solution of ammonium oxalate for 1 hour at 80°C. on a steam bath. Loose core test was used. Straw was washed as above.	A small amount of shive present. Fibre was harsh and dry. 8% loss in weight was noted after retting and air drying. C.
5.	H.T.Johnston	Same as #4 except straw was treated for 3 hours in an 0.5% solution of sodium oxalate.	Some shive was present. Fibre was dry. 10.1% loss in weight. C.-D.
6.	H.T.Johnston	Same as above except an 5% solution of sodium oxalate was used.	Some shive present. Fibre dry. 6% loss. C.-D.
7.	H.T.Johnston	Greenscutched fibre from Maxwell 1945 straw was treated with 0.5% ammonium oxalate for one hour at 80°C. on a steam bath. Washed as above.	A small amount of shive was present. Fibre dry. C.
8.	H.T.Johnston	Same as #7 except treatment was given for ½ hour.	Considerably more shive was present. Fibre harsh and dry. D.
9.*	H.T.Johnston	Same as #7 except treatment was given for 1½ hours.	A very small amount of shive was present. Fibre dry. 17.8% loss in retting. B.
10.	Dr. Fred Smith (66) at University of Minnesota	65 grams of Maxwell's green-scutched fibre was first treated in a water boil for one hour, followed by treatment for one hour at room temperature in one litre of calcium hypochlorite (0.35% available chlorine). Then the fibre was subjected to a two hour treatment at 66°C. in a solution made up of 300 ml. of 2% sodium hydroxide and 300 ml. of 3% sodium sulphite. Finally, the fibre was rinsed in hot water.	Fibre was bleached somewhat. Some shive was present. Fibre dry. C.

Ret No.	Originator	Retting Procedures	Results
11.	A.M.Munro	Maxwell's straw was treated with 0.02N oxalic acid solution for 2 hours at 100°C, followed by treatment for the same time and at the same temperature with 2% sodium hydroxide. Ratio liquor to straw, 15:1.	A large amount of shive was present. D.
12.	H.M.Flax Establishment, Norfolk, England	Green-scutched Maxwell's fibre was treated with 0.21% sodium hydroxide at 60-65°C. for ½ hour. Liquid to straw, 38:1. Fibre was then washed in cold water, followed by steeping in 0.26% (by volume) cold glacial acetic acid for 10 minutes.	A large amount of shive was present. D.
13.		Maxwell's straw was boiled for ½ hour in 1.25% sodium hydroxide. Then it was washed first in hot water, then in cold.	A small amount of shive was present. The fibre was lustrous and not harsh as in previous rets. B.-C.
14.		The same treatment as in #13 was carried out except time was increased to 40 minutes.	Slightly over-retted. B.-C.
15.*		The same treatment as in #13 was carried out except time was increased to 35 minutes.	The fibre was free from shive and had a lustrous appearance. B.
16.		Green-scutched fibre was boiled for 25 minutes in an 0.5% sodium hydroxide solution.	A small amount of shive was present. Fibre was harsh and dry. C.

The primary purpose of chemical retting is to loosen the fibre in the straw by solution or hydrolysis of cementing substances (pectins and gums) so that the fibre may be separated easily in subsequent mechanical operations. This purpose was achieved quite successfully with 0.5% ammonium oxalate and with 1.25% sodium hydroxide as exemplified in rets #3, 9, and 15. But it should be noted that while the chemical rets achieved in some cases the fairly complete removal of shive, the fibre finally obtained with even the most successful chemical rets lacked the degree of softness and pliability of water and dew-retted fibre. Nevertheless, such rets, which could be performed rapidly in the laboratory on large numbers of small samples, would serve as a quick method of retting the

flax straw obtained from the field; thus, both the yield and quality of flax fibre from a large number of experimental plots could be quickly appraised.

II. Chemical Studies of Fibre.

The results of the analyses have been calculated to a moisture free basis and are given in Table II.

TABLE II

Chemical Composition of Fibre

Description	% of Dry Matter					
	Lignin	Cellulose	Ash	Ether Soluble	$CHCl_3$ Soluble	Alcohol Soluble
B.C.-1--Water Retted	4.29	86.36	0.65	2.20	0.95	0.89
C 2	3.80	85.65	1.14	1.91	1.02	1.10
C 3	4.48	86.19	0.79	1.59	0.79	1.10
C 4	4.77	82.54	1.22	2.00	0.91	1.09
Ottawa-C 1-Dew-Retted	5.24	82.13	1.48	1.68	0.83	1.81
C 2	5.88	80.37	1.52	1.84	0.95	2.36
C 3	5.38	84.29	1.63	1.54	0.86	1.75
C 4	6.05	83.24	2.08	1.52	0.82	1.75
1945 Crop						
Maxwell's water-retted	4.87	85.72	0.86	1.73	1.14	0.95
1946 Crop (C4)						
MacVicar, water-retted (C3)	5.28	87.04	1.15	2.20	1.28	1.10
1945 Crop						
Maxwell's Dew-Retted (C2)	4.50	82.93	1.18	1.63	0.82	1.44
Univ. Dew-Retted (1946 Crop)(C4)	6.17	75.30	1.68	1.88	1.31	3.23
Chem. Ret #3	4.67	86.66	1.07	1.90	0.99	0.76
Chem. Ret #9	4.29	84.95	1.61	1.63	0.60	0.52
Chem. Ret #15	3.77	88.09	2.19	1.34	0.67	1.04

It can be seen from the table that water-retted fibre contained less lignin than did the dew-retted fibre. It was found that the lignin of the chemically retted fibre was of the same order as that of the water rets. With respect to cellulose, the water-retted fibre contained more than did the dew-retted fibre, and again the chemically retted fibre tended to be similar to water-retted fibre. It was of interest that the University sample of very poor quality dew-retted fibre was particularly low in cellulose. The ash

content of water-retted fibre was less than that of dew-retted fibre, and here the chemically-retted fibre tended to agree with the dew-retted fibre.

With regard to ether, chloroform and alcohol, water-retted fibre was found to be higher in ether soluble material, and lower in alcohol soluble material than dew-retted fibre.

Summing up the differences among the three types of rets, water-retted fibre was lower in lignin, ash, and alcohol soluble material, and higher in cellulose, and ether soluble material than was dew-retted fibre, while the chemically-retted fibre more nearly approached water-retted than dew-retted fibre in composition.

There appeared to be no significant relationship between grade of fibre and lignin, cellulose, ash, or extracted material.

III. Pressley Tests.

It became obvious from preliminary testing of tensile strength of flax fibre with the Pressley machine that large variations existed between breaks on the same sample, that there was an operator difference, a difference between jaws or clamps, and that there appeared to be a difference from day to day. Experiments were designed to investigate these differences.

Experiment 1.

Investigation of the Significance of Differences between Operators and between Days.

Two operators, H and A, using a sample of Manitoba dew-retted and a sample of Manitoba water-retted flax, each carried out seven breaks on each sample daily for ten days.

The operators alternated so that, on one morning, operator H did seven breaks on water-retted fibre, and operator A did seven breaks on the same fibre; in the afternoon operator H did seven breaks on dew-retted fibre, and operator A did seven breaks on dew-retted fibre. The following day, the order of operators and fibres was reversed. This procedure was carried out for ten days. Data from all clean breaks were retained, so that there were finally 280 values for study.

TABLE III.

Summary of Data (Experiment 1).

	Operator "H" (Means of 7 breaks)		Operator "A" (Means of 7 breaks)	
Day	Water-Retted	Dew-Retted	Water-Retted	Dew-Retted
1	7.83	7.83	6.76	7.73
2	6.03	4.32	4.90	5.10
3	6.79	7.58	6.55	5.50
4	5.39	6.92	4.07	4.88
5	10.83	8.51	6.61	5.49
6	6.75	7.93	6.92	5.79
7	8.47	10.90	10.25	5.83
8	8.90	8.97	5.35	7.95
9	7.40	8.94	8.07	7.31
10	6.19	7.99	5.70	6.37
Means	7.46	7.99	6.52	6.20

Analysis of Variance (Experiment 1).

Source	Sum of Squares	Degrees of Freedom	Variance
Days (1-10)	359.7307	9	39.9701
Treatments (rets)	0.7865	1	0.7865
Operators (H & A)	130.9997	1	130.9997
Interaction (Days x Treatments)	66.2222	9	7.3580
Interaction (Days x Operators)	64.2145	9	7.1349
Interaction (Treatments x Operators)	12.7203	1	12.7203
Interaction (Days x Treatments x Operators)	118.8432	9	13.2048
Error (within groups of 7)	623.0777	240	2.5962
Total	1,376.5948	279	

The differences between days were significant in relation to "Error" but not to the "triple interaction". The difference between operators was highly significant and was the largest source of variance.

An effort was made to reduce the variation between operators. H & A worked together for several days, and both agreed upon and practised an

apparently identical technique.

Experiment 2.

Investigation of the Significance of Differences between Operators, between Days and between Jaws.

A second experiment was conducted on Manitoba water-retted fibre in the same manner as the one above, with the exception that each operator carried out ten breaks per day on each of two sets of clamps; five breaks on each pair of jaws in the morning by each operator and the same procedure in the afternoon. The experiment continued for five days. Samples were selected between the weights of 0.3 and 0.5 mg. as a study of the previous data showed that there was not a high correlation between breaking strength and weight of sample.

TABLE IV.

Summary of Data (Experiment 2).

	Operator "H" (Means of 10 breaks)		Operator "A" (Means of 10 breaks)		Means by
Day	Clamps #316	Clamps #323	Clamps #316	Clamps #323	Days
1	7.07	10.62	6.66	10.24	8.65
2	7.62	10.20	7.17	8.22	8.30
3	8.43	8.94	7.60	8.82	8.44
4	8.44	8.65	8.13	7.86	8.27
5	8.49	8.68	6.31	8.74	8.05
Mean	8.01	9.42	7.17	8.78	8.34

Analysis of Variance (Experiment 2).

Source	Sum of Squares	Degrees of Freedom	Variance
Days (1-5)	7.7310	4	1.9328
Clamps (#316 & #323)	113.1910	1	113.1910
Operators (H & A)	27.2617	1	27.2617
Interaction (Days x Clamps)	71.4521	4	17.8630
Interaction (Days x Operator)	5.5364	4	1.3841
Interaction (Clamps x Operator)	0.4901	1	0.4901
Interaction (Days x Jaws x Operator)	19.8792	4	4.9698
Error (within groups of 10)	748.6228	180	4.1590
Total	994.1643	199	

When F values were calculated using either the "error" or the "triple interaction", the difference between clamps was highly significant. The difference between operators had been largely eliminated as compared with the previous experiment. It was significant when compared to the "error variance". The difference between days was negligible, which was very encouraging.

The "error", however, after eliminating days, clamps, and operators, was still much too large for a routine laboratory technique. It would be necessary either to do a large number of breaks on each sample or else to reduce greatly the error if the instrument were to be used to measure small differences between lots of fibre.

Further Analysis of the Data from Experiments 1 and 2.

The data from Experiment 1 and Experiment 2 were further studied to determine whether or not breaking strength and weight of sample were closely related. The "Covariance Technique" of Goulden (32) was used. In Table $\overline{V}$, x = weight of sample and y = breaking strength as read from the Pressley Instrument.

Experiment 1.

There were significant differences between days and between operators when compared with the error variance. The differences, however, were not significant when compared with the triple interaction variance.

Experiment 2.

A similar study of the data from Experiment 2 showed a highly significant difference due to clamps and a barely significant difference due to operator. There was no significant difference due to days.

TABLE V.

Experiment 1.

Analysis of Covariance
Operators H & A.

	D.F.	$\leq x^2$	$\leq(xy)$	$\leq(y^2)$	byx	byx $\leq(xy)$	$\leq y1^2$	D.F.	rxy
Days (1-10)	9	3.6096	3.1744	21.5739	0.8794	2.7916	18.7823	8	0.3597
Treatments (D & W)	1	0.4561	2.2414	11.0168	4.9143	11.0149	0.0019	0	0.9999
Operators (A & H)	1	2.8603	4.2086	6.1925	1.4714	6.1925	0.0000	0	1.0000
Interaction (Days x Treatments)	9	0.8564	3.1830	45.9686	3.7167	11.8303	34.1383	8	0.5073
Interaction (Days x Operators)	9	0.7465	5.0275	67.1605	6.7348	33.8592	33.3013	8	0.7100
Interaction (Treatment x Operators	1	0.0009	-0.0196	0.4305	-21.7778	0.4268	0.0037	0	-0.9949
Interaction (Days x Treatment x Operators)	9	0.5775	2.0184	45.8630	3.4951	7.0545	38.8085	8	0.3922
Error (within groups of 7)	240	10.4485	61.3304	629.2949	5.8698	359.9972	269.2977	239	0.7563
Total	279	19.5557	81.1641	827.5004	4.1504	336.8635	490.6369	271	0.6380

Test of Significance.

	D.F.	$\leq x^2$	$\leq xy$	$\leq y^2$	byx	byx$\leq(xy)$	$\leq y1^2$	D.F.
Days + Error	249	14.0581	64.5048	650.8688	4.5884	295.9738	354.8950	248
Treatment + Error	241	10.9046	63.5718	640.3117	5.8298	370.6109	269.7008	240
Operators + Error	241	13.3088	65.5390	635.4874	4.9245	322.7468	312.7406	240

TABLE V (cont'd)

Test of Significance

	D.F.	S(Sq.)	Variance	F.	5% Point
Days + Error	248	354.8950			
Error	239	269.2977	1.1268		
Diff. = Days	9	85.5973	9.5108	8.44	1.80
Days	8	18.7823	2.3478		
Diff. = b_e-b_{days}	1	66.8150	66.8150	59.30	3.89
Treatment + Error	240	269.7008			
Error	239	269.2977	1.1268		
Diff. = Treatments	1	0.4031	0.4031	Not Significant	
Treatments	0	0.0019			
Diff. = b_e-$b_{treat.}$	1	0.4012	0.4012		
Operators + Error	240	312.7406			
Error	239	269.2977	1.1268		
Diff. = Operators	1	43.4429	43.4429	38.55	3.89
Operators	0	0.0000			
Diff. = b_e-$b_{operators}$	1	43.4429	43.4429	38.55	3.89

TABLE VI

Experiment 2

Analysis of Covariance
Operators H & A

	D.F.	ξx^2	ξxy	ξy^2	byx	byxξ(xy)	$\xi y.1^2$	D.F.	rxy
Days (1-5)	4	0.0564	0.3684	3.5039	6.5319	2.4064	1.0975	3	0.8288
Jaws(#316 #323)	1	1.0105	-0.3298	10.3467	-31.4095	10.3589	-0.0122	0	-1.0000
Operators (H & A)	1	0.0300	-0.1324	0.5843	-4.4133	0.5843	0.0000	0	-1.0000
Interaction (Days x Jaws)	4	0.0052	-0.1548	8.5826	-29.7692	4.6083	3.9743	3	
Interaction (Days x Operators)	4	0.0347	0.2584	2.7361	7.4467	1.9242	0.8119	3	
Interaction (Jaws x Operators)	1	0.0000	-0.0014	0.1463	0.0000	0.0000	0.1463	0	
Interaction (Days x Jaws x Operators)	4	0.0077	0.0229	3.5813	2.9740	0.0681	3.5132	3	
Error (within groups of 10)	180	0.9252	3.9338	116.7356	4.2518	16.7257	100.0099	179	
Total	199	1.0697	3.9651	146.2168	3.7067	14.6974	131.5194		0.3170
Days + Error	184	0.9816	4.3022	120.2395	4.3828	18.8557	101.3838	183	
Jaws + Error	181	0.9357	3.6040	187.0823	3.8517	13.8815	113.2008	180	
Operators + Error	181	0.9552	3.8014	117.3199	3.9797	15.1284	102.1915	180	
Days + Triple Interaction	8	0.0641	0.3913	7.0852	6.1045	2.3887	4.6965	7	
Jaws + Triple Interaction	5	0.0182	-0.3069	13.9280	-16.8626	5.1751	8.7529	4	
Operators + Triple Interaction	5	0.0377	-0.1095	4.1656	-2.9045	0.3180	3.8476	4	

TABLE VI (cont'd)

Experiment 2

Analysis of Covariance
Operators H & A

Test of Significance

	D.F.	S(Sq)	Variance	Value F	5% Point
Days + Error	183	101.3838			
Error	179	100.0099	0.5587		
Diff. = Days	4	1.3739	0.3434		
Days	3	1.0975	1.3658	Not significant	
Diff. = $b_e - b_{days}$	1	0.2764	0.2764		
Jaws + Error	180	113.2008			
Error	179	100.0099	.5587		
Diff. = Jaws	1	13.1909	13.1909	23.61	3.89
Jaws	0	-0.0122			
Diff. = $b_e - b_{jaws}$	1	13.2031	13.2031	23.63	3.89
Operators + Error	180	102.1915			
Error	179	100.0099	.5587		
Diff. = Operators	1	2.1816	2.1816	3.9048	3.89
Operators	0	0.0000			
Diff. = $b_e - b_{operators}$	1	2.1816	2.1816	3.9048	3.89
Days + Triple Interaction	7	4.6965			
Triple Interaction	3	3.5132	1.1711	Not Significant	
Diff. = Days	4	1.1833	.2958		
Days	3	1.0975			
Diff. = $b_e - b_{days}$	1	0.0858	.0858		
Jaws + Triple Interaction	4	8.7529			
Triple Interaction	3	3.5132	1.1711	Not Significant	
Diff. = Jaws	1	5.2397	5.2397	4.4742	10.13
Jaws	0	-0.0122			
Diff. = $b_e - b_{jaws}$	1	5.2519	5.2519	4.4846	10.13
Operators + Triple Interaction	4	3.8476			
Triple Interaction	3	3.5132	1.1711	Not significant	
Diff. = Operators	1	0.3344	.3344		
Operators	0	.0000			
Diff. = $b_e - b_{operator}$	1	.3344	.3344		

In Exp. 1 there were highly significant differences due to days and operators. The difference due to days had been eliminated in Exp. 2, and that due to operators had been greatly reduced.

The correlations between weight of sample and breaking strength were highly significant for both experiments 1 & 2, the coefficients being + 0.6380 and + 0.3170 respectively. These correlations, however, were not great enough to justify the use of the equation: Index = $\frac{\text{Breaking Strength}}{\text{Weight of Sample}}$, particularly because the ratio of Weight of Sample to Breaking Strength decreased as the sample weight increased. In fact, there was a significant negative correlation between weight of sample and Index as calculated.

Dr. Paul Larose (42) of the Textile Section of Chemistry Division of National Research Council believed that the chief reason that decreasing indices occurred with increased size of sample was that there was failure in gripping all the fibres in the jaws in such a manner that they were initially all under the same tension. A re-designed pair of jaws exerting a stronger pressure at the edge than further in the jaw might eliminate this discrepancy.

It was concluded, first, that with the Pressley apparatus, the relationship: Index= $\frac{\text{Breaking Strength}}{\text{Weight of Sample}}$ did not hold for flax fibre; it was found necessary either to use a uniform weight of sample (which required discarding many results because the weight cannot be determined until the break has been completed) or to use a regression equation in the calculation of the Index. Secondly, it was found, as seemed apparent in preliminary testing, that different jaws or clamps contributed to the difference in indices observed. Thirdly, daily and operator variations were eliminated with practice in the use of the Pressley machine and in carrying out the procedure for tensile strength [illegible] in an identical manner.

Experiment 3.

Relative Humidity and Breaking Strength.

Magnesium Acetate $[Mg\ (C_2H_3O_2)_2 \cdot 4H_2O]$ in a supersaturated solution with water, maintains a relative humidity of 65% at 20°C. with the humidity gradually decreasing with rise in temperature. The breaking strength determinations for Exp. 1 were done in the summer when the laboratory temperature ranged from 25°C. to 30°C. The samples, conditioned over Magnesium Acetate, were tested at laboratory humidities, which for the ten day period of Experiment 1 ranged from a low of 40.7% to a high of 63%. There was no correlation between the relative humidity and the daily mean of the 28 Indices when the data were examined statistically.

Experiment 4.

Tensile Strength Tests on Chemical Rets.

The Pressley Strength Tester, in spite of its limitations, was used in assessing the relative tensile strengths of fibre from certain of the chemical rets. The work was carried out by one operator using one set of clamps. Twelve breaks were carried out on each sample and one break was completed on each of the five samples before a second was commenced, and so on. Thus variations due to operator, clamps, and days were eliminated. Sample weights were kept within 0.3 to 0.5 mg. The summary data follow with the necessary difference for significance (P = 0.05) between means.

TABLE VII.

Ret No.	Treatments	"Index" (Means of 12 breaks)
7	Water-retted	9.21
3	Ammonium Oxalate, 0.5%	10.31
4	Ammonium Oxalate, Saturated	9.41
5	Sodium Oxalate, 0.5%	9.24
6	Sodium Oxalate, 5.0%	8.10
	Necessary Diff. (P = 0.05)	1.64

The water ret was the standard Pilot Flax Mill Ret at 76°F. The Index showed Ret #3 to be significantly stronger than Ret #6. There were no significant differences between the remaining rets.

Experiment 5.

Tensile Strength Tests on Linen Yarn.

The Searle Grain Company, Ltd., Winnipeg, Manitoba, sponsored one-half acre plots of fibre flax at ten points in the four Western Provinces in 1944. The pulled flax was sent to Quebec to the Dept. of Home Economics and Handicrafts where it was dew-retted, broken, and scutched. Samples of these fibres were sent to the Linen Industry Research Association in Ireland where they were spun into yarn of eighteen lea size. The yarn samples were returned to the Searle Grain Company along with a sample from medium quality Irish flax. Major H. G. L. Strange very kindly made the yarn samples available for study along with test data from the L.I.R.A. The following table compares data obtained on the Pressley Cotton Fibre Strength Tester with Irish data obtained by breaking a 36" length on a single thread tester.

TABLE VIII.

	L.I.R.A.			Portage la Prairie		
Source of Flax	Actual Lea	Mean Breaking Load Oz.	Yarn Quality No. (Lea x Strength)	Mean Breaking Strength (24 breaks) (lbs.)	Index	Yarn Quality No. *
Irish dam retted flax	16.0	92.7	1480	5.74	4.71	1469
Creston, B.C.	16.2	88.3	1430	5.75	4.80	1490
Westlock, Alta.	15.0	93.7	1405	6.00	4.65	1440
Lebret, Saskatchewan	11.2	73.2	865	4.58	2.63	821
Arbourg, Manitoba	16.2	68.4	1110	5.66	4.72	1467
Necessary Difference**					0.60	

*Breaking Strength x 16 x lea
**Necessary difference for significance (P = 0.05)

The fibre from Lebret, Saskatchewan, was short and hand grading indicated that it was weak and inferior in quality as compared with other samples. It was of interest to note that this observed lack of quality was reflected in the yarn.

The yarn quality numbers as determined with the Pressley Strength Tester were of the same order as those determined on a 36" length with the exception of the Arbourg sample. The fibre from Arbourg was shorter than that from Creston or Westlock and did not appear to be evenly retted. A break on a 36" length of yarn spun from short fibre would reflect this failing in length, while a break on the Pressley machine requiring such a short length of yarn (0.464"), provided the fibre itself were strong, would not indicate the shortness of the fibre. Thus the differences between the Arbourg values obtained with the Pressley and with a 36" break may be attributed partly to the shortness of the fibre. The yarn from Lebret fibre was significantly weaker than the remaining yarn, and among these there were no significant differences in indices.

Discussion

One of the main objectives of this thesis was to find an unbiased scientific method of appraisal of the quality of flax fibre. As far as the chemical analyses have been carried, there does not appear to be a significant relationship between quality of fibre and the chemical constituents that were determined. It is probable that macro analyses of this nature were not selective enough to detect certain chemical constituents or properties which may be responsible for the quality of flax fibre as the trade knows it. These investigations are preliminary. A more detailed separation of the various types of cellulose, lignin and lipid-like constituents might reveal particular substances that are associated with a high quality of flax fibre. The large amount of experimental evidence that has accumulated in recent years as a result of experienced and intensive research in fibre chemistry, has indicated that the physical properties of fibres, which are responsible for those criteria which are usually associated with a high quality fibre, have their basis in a particular chemical configuration and composition. It should be the aim of future research in finding a chemical test of flax fibre quality, to attempt to discern as far as possible the exact chemical constituents of the fibre as well as those intimately associated with it and the nature of the association. The analyses reported in this study represent a first step in this direction.

Pending future developments in chemical analysis of flax fibre and on the basis of the tensile strength tests herein reported, one of the most promising approaches to flax fibre grading with respect to strength may lie in the successful application of methods similar to that of the Pressley Fibre Strength Tests.

But there are other considerations such as length, lustre, softness, cclour, freedom from extraneous matter, which the professional grader uses to identify high quality fibre. The physical tests with the Pressley machine, performed in the course of this investigation, were simply a method of finding a scientific test of fibre strength. Attempts should be made to find other physical measurements of the properties sought in high grade flax.

With respect to the physical measurements of fibre strength, there exist certain difficulties and complications which must be overcome before the Pressley Fibre Strength Tester can be used as a universal test of fibre strength. These complications have been described previously. The greatest obstacle exists in the method by which the fibre is gripped when stress is applied. When this difficulty is circumvented, it is possible that the Index used, namely $\frac{\text{Breaking Strength}}{\text{Weight of Flax}}$, will provide a valid test of fibre strength.

With respect to chemical retting, it was found that certain types of chemical retting produced fibre which was not too greatly inferior to that produced by water and dew rets. The rapidity with which flax may be retted conveniently in a chemical operation both in commercial practice and in preparation and processing of sample flaxes from experimental plots, would suggest that this chemical retting may supplant eventually the age-old water and dew retting processes. This has already been accomplished successfully on a laboratory scale in the United States.

An elaboration of the methods of chemical retting and of testing of flax fibre quality will do much to expedite the rapid development of flax industry in Canada.

Summary

An attempt was made to develop a chemical and physical method of testing flax fibre quality.

Methods are described to ret flax chemically and to test the fibre produced with the methods of testing that were investigated. Flax of fair quality has been produced in the laboratory by chemical retting with ammonium oxalate and with sodium hydroxide.

There appeared to be no significant relationship between flax fibre quality and quantity of cellulose, lignin, ash, and ether, alcohol and chloroform soluble constituents.

Physical tests of fibre strength were carried out with the Pressley Fibre Strength Tester and the results were examined statistically. Because of the inadequate method of gripping of the flax fibre for the strength tests, no conclusions can be drawn as yet from tests made by this machine.

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