

AN ABSTRACT OF THE THESIS OF

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Abstract Approved: -----
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The retting of flax requires from several days to a week or more by present methods. This necessitates the holding over of one season's crop until the following summer, thereby introducing expensive risks, large capital investments, and increased storage costs. These costs have tended to hold back the industry in this state. The problem of retting is therefore one of economic importance, and any improvement that could be effected would aid in reducing the cost of production and would probably result in an expansion of the entire industry.

A number of experiments were conducted in an attempt to produce a better quality of fiber in a shorter time. Most of the work was based on the hypothesis that any improvement which might be effected would involve the use of chemicals to degum decorticated fiber. Samples of decorticated fiber were water retted to find out whether breaking of the unretted flax straw weakened the fibers. Dew retting tests were conducted in closed chambers with controlled temperature and humidity in an attempt to produce a high quality fiber at a low cost.

Various chemical solutions were used at different concentrations and temperatures for varying lengths of time in an attempt to find one or more that would effectively ret flax. Acids, bases and inorganic salts were not satisfactory. Soaps and ammonium and sodium oxalates gave the most satisfactory results, the former producing a higher quality fiber than the latter. Difficulty was encountered in removing the adhering soapy materials from the degummed fiber. Washing with warm water gave better results than with cold water, dilute acid, alkali or ammonium oxalate solutions. The amount of warm water necessary to remove most of the adhering soapy materials was prohibitive from an economic standpoint.

Soaps yielded a creamy-white to white fiber of fairly good quality. Degumming was not uniform, the fiber often retained streaks of darker colored fiber. The fiber strength was about equal to that from water retted decorticated fiber.

Water retted decorticated fiber was darker colored, more harsh and weaker than fiber from water retted straw. This supports the view that decortication of unretted straw lowers the quality of fiber. No standard test can be used to determine accurately when retting is complete thereby making it difficult to stop the process at the right time.

Dew retting under controlled conditions of humidity and temperature gave unsatisfactory results. Retting was not uniform and a darker colored fiber was produced. The process required at least a week longer than natural warm water retting in addition to requiring more labor and expense.

FLAX RETTING

by

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FLAX RETTING

Introduction

The retting of flax consists of breaking down the pectic substances that bind the fiber bundles to the surrounding tissues and to the central woody core of the flax stem. Retting is an important step in the production of flax fiber. The removal of fiber from flax stems is an expensive process and requires several days to a week or more by the methods used at present. These methods make it impossible for inexpensive plants or small retting establishments to ret large quantities of flax straw. This necessitates holding over one season's crop until the following summer, thereby introducing expensive risks, large capital investments, and increased storage costs. These costs have tended to hold back the industry here in Oregon. The problem of retting is, therefore, one of economic importance, and any improvement that could be effected would aid in reducing the cost of production and would probably result in an expansion of the entire industry.

With this in mind, a number of experiments were conducted by the writer in an attempt to produce a better quality of fiber in a shorter time. From a review of literature it was apparent that a large amount of work had been done along this same line and that any improvement which might be effected would probably involve the use of chemicals to degum decorticated fiber. A large portion of the experimental work was directed at the possibility of retting with

chemicals. In addition, water retting of decorticated fiber and dew retting of flax straw under controlled conditions of temperature and moisture were conducted in an attempt to cheapen the unit cost of production.

To familiarize the reader with flax retting in general and to enable him to better understand the experimental work reported in this paper, a number of factors affecting the quality of straw, the retting process, and the subsequent drying of the retted material are discussed previous to the presentation of the experimental data. A large portion of this preliminary material is based upon research work carried on by many investigators, largely European.

Literature Review

The ancient Egyptians were able to ret their flax in the warm waters of the Nile. This led to the adoption of retting in a slow current of water without any necessity for considering the cost of the process. At that time it is probable that little was known of the structure or chemical composition of the flax plant, or of the minute organisms that were responsible for the loosening of the fibers from the woody portions of the flax stem. With the advancements of science, workers have endeavored to obtain an accurate knowledge of all the processes of the plant during growth and in all its stages of preparation up to the time the fiber is ready for market. Chemistry, plant physiology, and bacteriology have all contributed to this study.

Extensive investigations of the factors affecting the retting process have been carried on only during the past two decades. During this time, however, great advances have been made towards increasing the knowledge of flax retting. With a portion of this knowledge at hand, a number of factors affecting the retting process will be discussed.

Factors Affecting the Quality of Straw

There are many factors affecting the growing flax plants. These factors determine the amount and quality of fiber that can be obtained from the straw and therefore are of importance when discussing the retting of flax. Some of the more important factors include climatic condition largely rainfall and temperature, soil conditions of which texture, structure, fertility, and drainage are most important, and various other factors such as rate and time of sowing, variety, and freedom from weeds.

These factors influence the height, stem diameter, tillering, and rate of growth which determine the quality of flax straw. Tall, medium sized stems without tillers yield a larger per cent of higher quality fiber than either large or small stemmed flax. Also large stems ret much more quickly than fine stems which often necessitates the sorting of the flax straw previous to retting so as to obtain a more uniform quality of fiber.

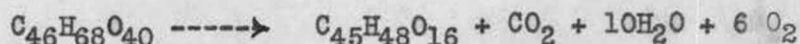
It has been found that fine stemmed, poor quality flax is often associated with dry, hot growing conditions while larger stemmed flax of higher quality is produced under cool, moist

conditions. This was found to be the case here in Oregon during 1930 and 1931. The straw produced in 1931, which was a relatively dry year, was fine stemmed, hard, and contained a small per cent of fiber while the year previous an unusual amount of moisture was present during the growing season and larger stemmed straw containing a higher per cent of fiber was produced.

Ripeness When Harvested

The stage of maturity of the flax straw at the time of harvesting has a marked effect on the retting process and upon the quality of fiber produced. Lazarkevitch (46) states that in Ireland, Oriental Flanders, and Holland, flax is generally harvested at green maturity, often being pulled early in the morning, deseeded later during the day by the aid of an iron comb, and placed in a retting pond or in the River Lys that evening. A very fine, but not extra strong fiber is obtained which is used in the manufacture of fine materials such as cambric and lace work. He and Robinson (59) state that flax harvested during the yellow maturity stage gives a medium fine, strong, elastic fiber while if allowed to mature fully before harvesting, it becomes lignified and the fiber loses its elasticity and separation is more difficult. A larger yield of tow is obtained from straw allowed to fully ripen before harvesting when it is worked up, due to a breaking of the fibers which have become lignified. Anderson (1), Ehrlich (27), and others have found that lignification interferes with retting. The latter considers that a conversion of pectin into lignin is caused by chemical and enzymic processes

during growth and aging, and may be represented by the following equation:



By harvesting flax straw before it was fully mature Robinson (59) found no statistically significant decrease in the quantity of fiber produced which is somewhat in accordance with the results obtained by Hutchinson (37), but not generally believed to be the case from commercial results. Several workers have reported on the effect of early harvesting on the vitality of the seeds among which might be mentioned Dillman (24), Barker and Eyre (8), Bredemann (14), Eyre and Fischer (28), and Opitz and Pander (54). In general it was found that when flax is harvested in the green stage of maturity most of the seed is sacrificed but if it is harvested in the yellow stage a large portion is recovered as it will complete its ripening during the following few days usually allowed for drying.

Eyre and Nodder (29) found green straw to ret much quicker than ripe straw but that the retting process must be stopped at just the right time; otherwise a serious weakening of the fiber bundles results. Also a larger amount of acidity is produced with the green straw which they attribute to be due to the presence in such straw of larger quantities of sugars and possibly free acids. This greater acidity, however, does not produce a harsh fiber but rather a very soft, pliable fiber which Robinson (60) rates as being of better quality than from straw harvested at any later stage of maturity.

Temperature of the Retting Water

The warm water retting process was developed by Schenk in 1846 in which the temperature of the retting water was raised up to 32° C. Previous to that time heat from the sun's rays was used, and except in warm areas and during the summer months of cooler areas, this source of heat was inadequate and prolonged the retting period. Since that time artificial heating of retting tanks has developed and is now being used to a large extent in most flax growing areas including Belgium where it is replacing a portion of the retting formerly carried on in the River Lys.

Eyre and Nodder (29), Huntley (36) and others found retting to be most rapid at 37° C. Above 40° C. the action of normal retting bacteria is markedly inhibited and other less active organisms "obtain the ascendancy" with the result that retting becomes very slow. Also below 20° C. the rate of retting decreases as the temperature falls.

In general it has been found that between 30-32° C. the most satisfactory results are obtained in ordinary water retting. Higher or lower temperatures may be used to promote retting by specific organisms; e.g., in the Carbone Process using the organism Bacillus felsineus, a temperature of 37° C. is required. The danger of over-retting is much greater at higher temperatures while with lower temperatures retting is so slow that it is often possible to dry samples of straw and work them up to determine when the straw is sufficiently retted. A drop of 10 degrees or more of the retting

water is liable to disturb the normal course of retting and set up abnormal fermentation characterized by the formation of considerable quantities of marsh gas (70).

Proportion of Straw to Water

The proportion of flax straw to water is believed to have some effect on the length of time necessary to complete the retting process and also upon the quality of fiber produced. However, this is not usually considered in ordinary tank retting or in stream retting to be a factor that should be varied with different lots of straw. Ruschmann (68) states that the proportion should range from twenty to eighty to one by weight of straw for different varieties to obtain satisfactory retting. It would seem more probable that differences between varieties would have a greater bearing on the quality of fiber produced than by varying proportions of straw to water. In canal and river retting to be discussed later it will be seen that a much greater proportion of water than even the eighty to one ratio is present and very good retting is obtained.

Retting Organisms

Many early workers studied the water of retting pools and ascertained the presence of different organisms. Van Tieghem (86), in 1879 carried on some early investigations on the microbiology of flax retting designating Bacillus amylobacter as being responsible for retting. The first real work of this kind reported, however, was that of Winogradsky (88) in 1895 in which ten different species were isolated by M. Fibres. Hauman (33) in 1902 probably was the first

worker to determine definitely that a number of organisms were responsible for retting. He found that B. mesentericus, B. subtilis, Streptothrix sp., and Pseudomonas fluorescens were able to ret flax efficiently. Hoffmeister, in 1910, as mentioned by Carter (15) added to our knowledge of flax retting bacteria by determining that Bacterium megetherium was least active while Bacillus subtilis, B. patrificus coli and B. fluorescens liquefaciens were most active in retting flax.

A number of other writers have reported on organisms responsible for retting among which might be mentioned Behrens (10), Stormer (28), Rossi (64), Wyant (89), Carbone (17,18,19), Ruschmann (67,68,69), Tanner (79), and Trevethick, et al (83). The findings of some of these workers will be mentioned later.

Of the more recent writers Omeliansky (53) believes that Granulobacter pectinovorum is responsible for retting but is accompanied by other bacteria having the role of favoring the retting process, hastening it and giving a better product by protecting the anaerobic retters from direct action of oxygen.

Makrinov (48) recently reports that pectic fermentation by the anaerobic organism Granulobacter pectinovorum does not yield fully oxidized compounds, the end products being butyric acid, a small amount of alcohol, and minute amounts of carbon dioxide and hydrogen. On the other hand, Pectinobacter amylophilum yields completely oxidized products, carbon dioxide and hydrogen, with about 25 per cent alcohol and formic acid. The explanation being

that with a greater oxidizing power the aerobe causes destruction of the plant pigments and extractives as well as bleaching of the fiber which do not occur in the presence of the anaerobe. Makrinov further states that in the retting of flax with Pectinobacter the fluid becomes more active if used over several times.

Stampa (77) has grouped the organisms responsible for retting into the following divisions:

1. Aerobes

- a. Group of Bacillus subtilis and mesentericus (B. mesentericus vulgatus, investigated for the purpose by Vignal; B. of Mermier; B. pecticus and others of the same type of Rossi; B. of Ruschmann; and B. of Trevethick, Robinson, and Snyder).
- b. Group of B. asterosporus (B. asterosporus Mayer Migula; B. macerans Schardinger; B. comesii Rossi; B. Kramerii Rossi and Carbone; Bacillus B of Makinov and Ciglio; Pectinobacter amylophilum Makrinov).

2. Anaerobes under certain circumstances and less strictly anaerobic.

These organisms even if capable of more or less aerobic life are usually active for retting in anaerobic conditions. They include a number of species and varieties belonging to the group of Bacillus amylobacter in the sense of Bredemann (Granulobacter pectinovorum and Granulobacter urcuphalum of

Beijerinck and van Delden--Plectridium pectinovorum of Stoermer, bacillus de Winogradsky)--the bacteria 1, 2, 3, 4, 5, and the bacillus 6 of Kayser and Delaval.

3. Strictly anaerobic.

This group includes for the moment only the Bacillus felsineus Carbone, the B. maymonei Carbone, and probably Plectridium amarillum Sordelli and Soriano, of which it is merely known that it does not attack cellulose, that it produces a less energetic retting than that of Bacillus felsineus, and that it imparts to mashed potatoes a bright yellow coloration.

4. Thermophilous retting organisms still unknown, but probably active in the simple country retting of textile plants such as is carried on in the hot springs of Bulicame at Viterbo in Italy.

Stampa states that each of the groups of microbes (and even, in part, each of the species) confers on the fiber certain characteristics. The aerobes producing the false retting which is very different from the true retting of the Italian country retting tanks; the Amylobacters being capable only of producing a poor and uncertain effect, while the strict anaerobes, in particular B. felsineus, act uniformly and actively, and impart to the fiber characteristics of the best country retting methods carried out under water.

Pure Culture Retting

Retting has been accomplished by using pure cultures of a number of organisms and several processes have been developed. Probably the first attempt to ret with a pure culture was by Deumaer and Deswarte, as mentioned by Bazzocchi (9), in which they used a culture of Bacillus amylobacter to ret flax with apparently good results. Previously, however, Carter (15) mentions that Doumier in 1899 advocated a method of retting in warm water to which had been added bacteria isolated from retting liquor. Bazzocchi also mentions that Hiltner and Stormer used cultures of plectridium pectinovorum and other organisms of secondary value while Makrinoff used Pectinobacter amylophilus in his process.

About this same time Rossi (64) and his collaborators started extensive investigations at the Institute of Agricultural Bacteriology in the Royal Higher School of Agriculture at Portici, Italy. In 1916 he introduced a process named after himself which received approval in parts of Italy and France. During the war, however, the flax industry was practically at a standstill and several of the newly established retting plants were destroyed. This prevented the widespread adoption of this new process which at first seemed to be a great improvement over any previous process.

The essential features of the Rossi Process are as follows:

1. Immersing the textile materials in ordinary water.
2. Raising the temperature up to 28 to 30° C., which was "optimum" for the bacillus or culture used.

3. Addition of a suitable quantity of Bacillus comesii in the bouillon, prepared by means of ferments.
4. Passing a current of air through the whole of the retting liquor all during the retting process by means of perforated horizontal pipes at the bottom of the vats.

The two outstanding advantages of this process were the shorter period necessary to complete the retting process and the elimination of danger of over-retting. The disadvantages are many so only a few of the more important ones will be given. Ruschmann (71) found that a very intense aeration, many times that specified by Rossi, was necessary which entailed expensive and inconvenient equipment and operation, such expenditure not being justified economically. The fiber was found to be more dull in appearance, harder and darker in color than that obtained in the anaerobic process.

Bazzocchi (9) got very unfavorable results using this process, the fiber being so gummy that it could not be used commercially. He found the percentage of tow to be high while the time necessary to complete the retting process with hemp, flax, and ramie was 139, 141, and 157 hours respectively.

The Carbone Process

Carbone (17) isolated from the mud of some of the retting pits in Bologna, Italy, an obligate anaerobic bacillus which he called Bacillus felsineus, which was capable of retting hemp and many other textile plants. The addition of this bacillus to the

retting solution and the accurate maintenance of a temperature of 37° C. formed the basis of this process. Stampa (77) states that cultures of this bacillus are prepared in the liquid form of "Felsinoyima," or in the powder "Felsinoyima secco." The dry Felsinoyima is 10 times more concentrated than the former, more easily transported, especially to great distances, and has better keeping qualities. He states that from 3 to 10 liters of ferment consisting of one-half liter of liquid Felsinoyima or 50 grams of the powder in 10 liters of solution is added per quintal of textile plant in warm water in which the textile plant has been placed for retting.

The advantages of the Carbone process as summarized by Tobler (80) are as follows:

1. Reduced time needed to complete the retting process.
2. Better color of fiber.
3. Production of less smell and acidity or improved conditions of the effluent water.

Disadvantages:

1. Need of inoculation with a culture of specific bacteria.
2. Preparation of inoculating material.
3. High temperature of retting solution.

Another disadvantage which has received some little criticism is the danger of over-retting which is a very important factor when the retting proceeds so rapidly.

Bazzocchi (9) found that fiber obtained from using Bacillus felsineus was equal to that of the natural warm water retting process being completed in 75 hours with flax, 94 hours with hemp, and 97 hours with ramie. He also found that the retting liquor could be used for two or more macerations, but which does not seem practical or at least has not aided greatly in the widespread adoption of the process by flax producing countries.

The Kayser Process

Kayser and Delaval (38) isolated 6 organisms from retting textile plants and named them Bacillus numbers I to VI. No. VI formed the basis for the Kayser process (British patent No. 208,397, Jan. 6, 1923) which was not used to any extent. The process consisted essentially of adding a culture of the above organisms to the retting mass at a temperature of 25-28° C. which accelerated the retting process requiring from 60 to 70 hours for completion. Muller (34) states that the retting goes so far and there is an accompanying flora developed which will destroy cellulose.

The using of pure cultures of specific organisms to ret flax has been reported on by other workers. Probably the most outstanding investigation carried on in this country on the characteristics of specific organisms and pure culture retting was performed by Trevethick (83). She isolated a large number of organisms from scutched fiber and after determining the morphological, cultural, and physiological characteristics of those which were active retters, carried on a number of tests to determine the retting ability

of specific organisms by using sterilized straw. She concluded that there were three organisms that qualified as flax "retters" and which belonged to Bergey's mesentericus-megatherium group.

Robinson and Snyder (63) found when pure cultures of efficient retting organisms, as isolated by Trevethick, were added in large quantities, that the retting process was hastened only slightly. They suggested that the control of acidity in the retting bath to be a more promising method of producing a quicker ret and a better quality of fiber.

In summing up retting with pure cultures it has generally been found that the organisms responsible for retting increase on the flax straw almost as strongly without inoculation as with inoculation of a pure culture. Muller (34) states that with retting under ordinary conditions, bacteria are always found on the flax stems in great amount and the additional handling of pure cultures is not worth the while. He does not believe that a new bacillus will be present in any greater amount when pure cultures are added than under ordinary retting conditions. Also, that no better quality of fiber can be produced, the only advantage may be a shortening of the retting process. This, of course, has discouraged the practices employed in many new processes whereby pure cultures of organisms are added to speed up the retting process. Of all the pure culture processes that have been developed, Carbone's process has given the most satisfactory results. Even

with this advantage Stampa (77) states that in 1930 there were only 2 retting establishments in Italy employing this process.

Using of Old Retting Liquor

Portions of the old retting liquor have served as a source of inoculum for the water of new rets for a long time. Robinson (58) states that the basis or principle of this process was invented by Mr. Constant Vansteenkiste of Ypres, Belgium, about the beginning of this century.

A Vansteenkiste ret consists in using up to 40 per cent of old retting liquor and the remainder of the water from small ponds containing old liquor from many previous rets. After a day of retting, a small portion of the retting solution is allowed to flow out and a small jet of fresh water is admitted causing a circulation of the retting solution. Muller (34) briefly describes the Cousinne process which was used in France to a limited extent consisting essentially in adding a portion of the old liquor to the new retting solution. Also, milk of lime is added mainly to neutralize the acidity that develops.

Another example of this type of retting was that of Lucas (British patent No. 339,808, Dec. 23, 1929) in which a quantity of liquid from a previous first stage ret was added to the new retting water at a temperature of 25° C. which was maintained for 12-24 hours. A portion of the liquor is then run off and may be used for the next batch, the tank being filled with fresh water and the

temperature slowly raised to 37° C., which temperature is maintained until retting is complete.

Old retting liquor is used in various parts of Europe but usually only in areas where fresh water is scarce. The usual practice is to allow the retting effluent to stand a period of time in small open ditches or ponds before being used over again. This allows the heavier particles to settle out and a partial purification of the retting solution by aeration.

Circulation and Renewal of the Retting Solution

A number of processes have been developed, especially since the World War, which involved the circulation of the retting solution. In several processes the retting liquor is slowly withdrawn from the retting tank and then returned after receiving some sort of treatment, largely purification.

The Thellier-Soenens method as described by Stampa (77), Muller (34), and others, consists in pumping the retting water which overflows through an outlet back into the bottom of the vat. The solution is aerated in its transit by fine bubbles that are obtained by compressing air between the walls of a porcelain cylinder or one of porous sandstone. This aeration and the maintenance of a temperature of 33° C. forms the basis of this retting process which is probably the most modern system of industrial retting of flax being used in Belgium and Spain to any extent at the present time.

A number of patents have been issued Summers for processes based on the circulation of the retting solution; e.g., U. S.

patent No. 1,497,808, June 17, 1924, involving the withdrawal of the liquor from the retting material to be filtered with the idea of neutralizing the action of putrefaction bacteria. The filtered liquor is then returned to the retting vat. In a previous patent he claimed that the proportion of useful to putrefaction bacteria was increased by circulating the retting liquor from the vats through a filter in which the insoluble products of fermentation, which acted as culture bacteria, were collected and removed.

Closely associated with circulating the retting liquor is the preliminary leaching of the flax straw previous to retting. Clark's process (U. S. Patent No. 1,808,593, June 2, 1931) consisted in circulating cold water through the flax straw and then fresh water at 30° C. being admitted to replace the cold water, the temperature being maintained by circulating the water from the tank through an external heater and back again. The object of the preliminary circulation of cold water was to leach out a portion of the soluble materials and thereby get a better ret with the subsequent warm water.

The Teuss process as mentioned by Ruschmann (71) consisted in a very slow percolation into the vat of fresh water or the daily addition of about one-tenth the total volume of the retting solution.

Demytt (22) describing the Oregon State Flax Industry's former system of retting states that the tanks of flax straw were filled with fresh water, allowed to soak 24 hours and

then turned into the circulating system until sufficiently retted, adding a specified amount of fresh water to keep the water in the tanks "sweet." He states that experience shows this system to have several weak points, for instance, the power bill for running the pumps amounted to twenty dollars per day or nearly one dollar per ton of retted flax straw; the fiber obtained was drier and somehow coarser; the extra expense of installing pumps, motors and pipes (which have to be renewed at least 50 per cent each season), also more steam was required to keep the ret at a given temperature. As a consequence, the circulating method was discarded and individual tank retting adopted. By adding just the right amount of fresh water at the proper time a softer and more lustrous fiber with a better feel is obtained at a greatly reduced cost.

The advantages of circulating the retting solution as given by Stampa (77) are as follows:

1. A more uniform retting of the flax straw because of the unequal distribution of the specific organisms naturally found on the plants themselves.

2. Dilution of accumulated by-products of the retting process some of which are merely toxic for the microbes on whose action retting depends while others are evil smelling or poisonous to the fauna of the streams into which the retting efflux is drained or affects the color or other qualities of the fiber.

Improvements introduced to eliminate the accumulation of by-products have been discussed by Stampa and others. Stampa's

suggestions together with the writer's are grouped into the following classification.

1. Chemical neutralization of the harmful products. Several methods have been proposed in this connection but without success on account of their high cost. Tobler (81), Baird and Duff (7), Kraus (42) and others have used alkalies, largely sodium and calcium carbonates directed particularly towards neutralizing organic acids produced.

2. Mechanical separation of these products, as well as substances which bring about their formation. Soaking of the straw in warm water before final immersion in the retting water tends to prevent the formation of fetid substances.

Eyre and Nodder (29) list the changes occurring during the first stage of the retting process as follows: 1. Expulsion of air from between the straws and from cavities within the straws, 2. absorption of water by the plant tissues and their resultant swelling and softening, and 3. the dissolving in the water of the various soluble constituents of the straw, such as salts, sugars and other carbohydrates, glucosides, tannins, certain nitrogenous materials and coloring matters, which account for a considerable proportion (often more than half) of the total loss in weight which the flax undergoes in retting.

Ruschmann (71) states that among the organic acids formed butyric acid predominates and imparts to the flax and retting water its very disagreeable odor. The bulk of this acid, however, is not

materially reduced by preliminary leaching as it owes its origin to the pectic acid fermentation during the principal biological phase later in the retting process.

The elimination of toxic substances already formed is secured, on the other hand, by replacing the contaminated retting solution by fresh water after decomposition has begun. Stampa states that this results in a more rapid retting, with a less unpleasant odor, and yields a cleaner fiber. This method is employed in the Vansteenkiste and Legrand processes which are being used to some extent in Belgium.

3. Purification of the water in the retting tank by aeration after which it is returned to the vats. The processes devised by Rossi, Ochmann, Thellier-Soenens and DeGeyter (French Pat. No. 704,656, Oct. 21, 1930) all involve the aeration of the retting solution. Robinson and Snyder (63) by passing air through a retting solution kept the acidity from going very high with the result that a slightly higher percentage of fiber was produced than from a stagnant water ret. Ruschmann (71) states that in addition to producing a stronger fiber, low acidity facilitates the biological self-purification of the retting effluents, which is brought about by bacteria, fungi, etc. In summarizing the advantages of the percolation system of flax retting he states that it has been shown very definitely that over-retting in spite of the increase of the retting organisms on the fiber, can be almost completely avoided.

Lys River Retting

Many writers have described flax retting in the River Lys in Belgium which has become world famous for its fine linens. An area adjoining Courtrai for a distance of about 18 miles along the River is devoted almost entirely to the retting of flax. The river itself in this area is approximately 100 feet wide, the sides being boarded up so as to facilitate the emptying and filling of the retting crates. Retting starts in the spring months when the water is yet quite cold, about 10° C., the retting process requiring about 3 weeks.

Later in the summer the water becomes warmer and retting is completed in a shorter time. It is the general practice to double ret; i.e., allow the straw to ret about a week, remove it from the river, set the bundles up in stooks to dry and when dry put them back in the crates to ret several days longer. This gives a much higher quality and a finer fiber than can be produced by a single retting. The crates which float along each side of the river bank hold slightly over a ton of dry straw and are rented to people for so much a crate per year. In renting a crate in which to ret flax, the man also obtains a strip of land bordering along and running at right angles to the water front upon which he may dry his straw after it has been retted. The rent varies but in 1930 it was 1000 francs per year (\$30.00). Large amounts of flax straw are sent to the vicinity of Courtrai to be retted from Northeastern France, Groningen, Holland and various parts of Belgium. Robinson

(58) states that although a large portion of the retting is carried on in the Courtrai district, only about 20 per cent is actually retted in the River Lys, the remainder being retted largely in factories with warm water tank retting while a small quantity is dew retted.

Channel Retting

Canal or channel retting, a system devised by Schneider, consists of a continuous process in which fresh flax bundles pass slowly through an artificial tank or canal in a slow current of water so that by the time they reach the other end of the tank they have retted sufficiently and are removed. This gives a continuous process which is very important in large scale operation especially if the straw is to be dried artificially.

The Feuillette system as described by Durant (26), Muller (34), Ruschmann (68) and others employed the Channel system of retting being modified to the extent that the crates containing the bundles of flax were raised out of the retting water intermittently, thereby aerating the flax straw. This encouraged the development of aerobic organisms which were believed to be the real retters while the activities of anaerobic organisms were suppressed. The straw was then centrifuged about 15 minutes, being washed at the same time, and then artificially dried. The drying process consisted in passing wagon loads of retted straw through a tunnel, the wet straw entering at the air outlet end. Each hour the 12 wagons were advanced one step towards the fan so that the flax was

subjected to the higher temperatures only after being freed of a large portion of its moisture. Ruschmann (72) in describing this process in Germany states that the main advantages of canal retting is that the process is continuous. A disadvantage, however, is found in the increasing acidity due to the fermentation of the refuse settling in the bottom of the tank, which also retards proper movement of the retting water.

Another very important disadvantage is that different lots of flax straw which vary as to stem diameter, maturity, soil and climatic conditions under which grown, cannot remain in the retting bath but a certain time and then must be removed in turn. This, of course, would tend to give less uniformity than if retted in individual tanks. Also, the expense of operating such a set-up as described in the Feuillette process would be prohibitive from an economic standpoint and could not possibly be adopted without disastrous loss to the parties concerned. This is probably the main reason that this process failed to gain widespread use.

Character of Water Used in Retting

The composition of water used in retting flax has been studied quite extensively by various investigators. It is a well known belief that flax retted in the River Lys in Belgium is of higher quality than similar flax retted in any other river or by any other method. Waters from this river have been studied carefully by many research workers. Snyder (76) concluded that flax fiber is influenced to a great extent by the quality of the water

used. He found water from the River Lys, in Belgium, to be significantly higher in the alkaline chlorides and carbonates than water from the River Cannon, in Minnesota. Muller's (34) analyses of the water from the River Lys between retting vats were somewhat higher than those of Snyder but the former did not include as many individual mineral determinations as did the latter.

Diakonov (23) analyzed water used at 13 places in the province of Pakov, Russia, and found that good retting waters contain more substantial sediment, more mineral substances, more iron, aluminum, chlorine, and nitrogen than do poor retting waters. On the other hand, he found poor retting waters to contain more calcium.

Eyre and Nodder (29) found retting to be slower in rain than in tap water which they attributed to a smaller quantity of alkalis in the rainwater and, therefore, more acidity developed. Also, they found the loss in weight of the straw to be less when rain water was used.

Wyant (89) found river water to give better results than tap, distilled or cistern water. She considers the large amount of sewage in the river water to be responsible for this difference.

Kranzlin (45) states that chemical analyses fails to yield information of great significance for the evaluation of a water supply for use in retting flax.

Addition of Salts to the Retting Solution

Chemical analyses of retting waters from various districts showed them to contain varying amounts of dissolved materials. This explained, in part at least, why the water from a certain locality or river was better for retting purposes than waters from other places. With this knowledge as a background attempts were made to accelerate retting by the addition of salts, sugars, urea, and other materials to the retting solution.

Carter (15) states that Blet advocated the using of urine to encourage the development of retting bacteria. Tobler (81) and Flieg (30) by adding 0.25 per cent urea to the retting water shortened the retting process to some extent and obtained a slightly better quality of fiber. In addition, the waste liquor from which the urea crystallizes makes a good nitrogen fertilizer. Hacker (32) states that urea is a recommended addition to the water used in flax and hemp retting.

Krais (41) and Hacker found small additions of sodium sulphite to the retting water gave fiber of an improved color but that the odor emitted was very objectionable. In the steeping of nettles, the former found retting to be accelerated in a 0.5-1.0 per cent solution of sodium bicarbonate. Other bicarbonates; e.g., those of magnesium and ammonium, were similarly effective; sodium carbonate, magnesium hydroxide were less so, and sodium, potassium, and calcium hydroxides were non-effective. The use of borax instead of sodium bicarbonate checked the retting process with flax, nettles, and

mallow. Barium chloride acted similarly to check the retting process. In addition, he found that grape sugar and molasses checked the retting process in tap water but had no harmful effect in 0.1 normal sodium bicarbonate solution. Eyre and Nodder (29) found the retting period to be markedly prolonged by the addition of glucose.

Johnson Process

The Johnson process (British patent No. 151,143, Aug. 20, 1919) is similar to those of Kraus and Biltz, being carried on between 27 and 35°, using from one-half to two kilograms of chalk per 100 kilograms of straw. Slow circulation and aeration of the retting liquor are essential as is also a preliminary leaching for 4 hours.

Hacker (32) found additions of sodium sulphide, soap, and turkey red oil to aid retting to some extent. Smith (75) by adding 0.05-0.10 per cent of ammophos or diamphos (commercial ammonium phosphate) markedly hastened the retting of jute, while nitrophoska was almost ineffective.

The addition of materials to the retting solution to encourage the development of bacteria has not proved effective enough in hastening the retting process to warrant their adoption. The function of the organisms responsible for retting is to feed on the pectins of the flax stems in order that a loosening of the fiber bundles will take place; otherwise, the organisms may flourish on food which is outside the flax stems with the result that no retting is accomplished. On the other hand, the addition of certain materials may act indirectly by suppressing the acids which are

formed and in this way aid the retting organisms in breaking down the pectic materials of the flax stem. Furthermore, certain toxic by-products may be neutralized. It is also possible that the addition of salts, urea, etc. might aid in the development of certain organisms which assist the main retters by protecting them in various ways.

Muller's (34) criticism of urea retting probably can also be applied to the addition of salts, etc., to the retting solution in which he states that the process is too expensive for the benefits derived from such practice.

Artificial Drying

Flax straw upon removal from the retting tank contains approximately 400 per cent water, most of which must be removed before the fiber can be separated from the stems. The removal of this excess water presents a difficult problem to solve as the usual practice is to set the flax bundles up in stooks to dry by heat from the sun's rays during the summer months. This requires from several days to a week or more under uncontrolled conditions and would not work effectively if retting was continued throughout the fall and winter months. Quick drying under controlled conditions would be very much desired in this case. Artificial drying machines have been constructed to dry the retted flax straw, thereby controlling the drying process.

Results were quite unsatisfactory at first because of the large amount of heat required to evaporate the excess water. To

overcome this drawback, pressing machines were developed but were not very successful because of the poorer quality of fiber that was produced when these machines were employed. An improvement, however, by Vansteenkiste (85) at Ypres, Belgium, of an elaminating machine to overcome some of the previous difficulties has resulted in some success. Their machine with rubber rollers, peels, softens and partly scutches the ribbon of fiber in addition to removing approximately one-third of the water contained in the flax straw plus tissue residues, acids and bacteria which, by their subsequent development, may be harmful to the fiber.

Ruschmann (71) states that the greatest objection to pressing is that the fiber strength is decreased. He, however, considers centrifuging equally effective in removing water and acids without injury to the fiber. Muller (51) states that it is doubtful if the rolling of retted flax has any advantages to justify its adoption.

A large number of processes have been developed which necessitated artificial drying of the retted material. De Swarte and Doumer (British patent No. 245,752) dried retted flax straw without removing it from the retting tank by passing warm air up from below. The amount of heat required by this method, however, would be prohibitive, which could not possibly warrant its adoption. A number of machines have been devised similar to the one used in the Feuillette Process but none have gained any widespread use to date.

Several investigators have reported on the effect of artificial drying on the quality of fiber. In general, the reports have

been unfavorable in that the quality of fiber was lowered by artificial drying. The fiber has a somewhat more harsh feel and a poorer color, the latter being attributed to the lack of any bleaching effect, such as the sun's rays, when the fiber is artificially dried.

In summarizing the advantages and disadvantages of artificial drying retted flax straw, the latter outweigh the former under the present methods of flax manufacture. A shorter length of time under controlled conditions is the outstanding advantage. Also, if decorticated fiber is degummed either by chemical or biological means, artificial drying of the retted material is more practical than natural drying out of doors.

Disadvantages of artificial drying include increased cost and probably lower quality of fiber. Not only would extra capital be necessary for additional equipment but the cost of operating this additional machinery would increase the unit cost of production considerably. Whether this increased cost of drying would offset the costs and risks involved in carrying over a crop of flax straw until the following year would depend on a number of factors. In addition to the extra equipment and cost of operation mentioned above for artificial drying, the using of an elaminating machine to reduce by one-third the amount of water retained by the freshly retted straw, cuts down both on the time and heat required to dry the straw. This, however, requires an extra handling of the retted straw which

according to Vansteenkiste (85) requires about six and one-half hours time for each ton of dry flax straw or an increase of at least a half cent per pound of scutched fiber.

With the ordinary method of drying out in the sun, no cost is necessary except the handling of the straw. Climatic conditions in the Willamette Valley of Oregon during the summer months are quite ideal for the drying of retted flax straw. The days are usually hot and sunshiny while the nights are relatively cool and without the precipitation of dew. This aids in a rapid drying of the retted material with a minimum opportunity for the subsequent development of aerobic cellulose decomposing organisms as is often the case in areas where summer rains, heavy dews or cloudy weather are prevalent. This lessens the need for artificial drying considerably unless retting is to be carried on throughout the whole year.

Ruschmann (71) states that the most important advantage of natural over artificial drying lies mainly in the removal of harmful acids by volatilization at low temperatures or by oxidation by organisms.

Retting Decorticated Fiber

Decortication consists in breaking the unretted flax stems so as to remove the roots and a portion of the cortex and woody core. It is possible to remove practically all of the woody material from the fiber but most of the epidermis and various materials in close association with the fiber bundles remains intact and cannot be removed except with extreme mechanical treatment. By removing a

large percentage of the woody material the bulk of material is greatly reduced per unit of fiber which is of great significance under certain conditions where space or volume of retting solution is a factor. In chemical retting the unit cost of production is reduced because of the much smaller quantity of solution needed to ret a given quantity of fiber. This, however, will be discussed later under chemical retting.

Numerous attempts have been made to ret decorticated fiber biologically, especially during the past few years. Probably the greatest amount of work along this line has been done by Watson and Waddell of Belfast, Ireland, who have been awarded a large number of patents in various countries. In addition to retting decorticated fiber, they have developed a number of processes in which the fiber is degummed after it has been hackled and made into twisted rove, claiming that a saving of at least 80 per cent of the tedious and technical preparation necessary in the ordinary process of linen production is eliminated (87). They claim that mechanical decortication, without retting, has been unsuccessful because the fiber remains coated with an insoluble gum, which does not macerate in the hot-water trough of the wet spinning frame, and prevents the fibers from being drawn apart. They also have a number of patents in which yeast is used to remove the gums on the rove. One of their processes consists in heating the water up to 60° F. and adding 10 per cent yeast, which completes the retting in approximately 8 hours time. The solution need not be changed for 14 days as the yeast is

a living procreative organism and keeps on retting so long as it is given fresh rove to feed upon. Alcohol is produced in such quantities as may form a profitable by-product.

To make better quality yarn, as regards color and strength, 2-10 per cent sulphurous acid is added to the bath after the rove is sufficiently retted, later being washed thoroughly with cold water.

They have advocated the retting of seed flax straw to obtain fiber which could be spun to low counts.

Lowry and Grant Inc., of New York, have been working along the same line as Watson and Waddell, having perfected and patented a number of machines for decorticating flax straw and final degumming of the processed fiber (47).

Muller (34), Ruschmann (68), and others have discussed a number of other processes including those of Starling, Kuhlmann, Michotte, Pritchard, Dunbar, and Jahren.

In the retting of decorticated fiber it is almost imperative that the degummed fiber be dried artificially as was discussed previously. It is much more economical to dry the fiber artificially because of the smaller quantity of water that it contains as compared to the unbroken flax straw. Also, in addition to the smaller quantity of heat necessary, the individual bundles of fiber cannot be set up in the form of stooks but must either be laid down or suspended from above.

The disadvantages of artificial drying as discussed previously, also, apply for the most part to the degummed fiber as with the straw. It has been stated and seems probable that artificial drying of decorticated fiber injures the quality of the fiber more than does drying of the fibers while in the flax stems, as the fiber is exposed in the latter case. This, however, probably can be overcome by regulating the temperature and humidity of the drying chamber.

Dew Retting

In general, dew retting gives an uneven and less valuable fiber than either stream, tank, or pit retting. This is probably due to the fact that under ordinary conditions only poorer quality straw is retted by this method. This form of retting has been practiced since ancient times and is still being used to a more or less limited extent in certain parts of the world. It is the cheapest form of retting and is adapted more to small retting plants or individual farmers or peasants. The prime factors governing the success of dew retting may be listed as follows:

1. Nature of the retting bed or soil on which the flax is spread out to ret.
2. Atmospheric conditions during the retting process.

Under ordinary conditions out of doors these factors cannot be controlled except under special circumstances which makes it all the more important to choose a suitable retting bed for the flax. It is almost imperative that the retting bed be located near the

retting plant as transportation costs limit the distance to which the flax straw can be moved.

With a satisfactory retting bed available, the atmospheric conditions influence the retting process greatly. Atmospheric conditions which are most favorable for dew retting include a suitable supply of moisture especially in the form of dew and dampness from the grass and soil below, both of these factors aiding in retaining the fluctuating moisture content of the flax straw. Warm nights and not extremely hot days are essential as often with great heat and intense solar radiation not only are vegetative forms of the retting organisms destroyed but also the resting spores.

Other factors affecting the retting process include the evenness and thickness that the flax straw is spread on the ground and the regularity in turning the flax straw. Machines have been perfected to carry on these operations so that very little actual hand labor is necessary. This aids considerably in some cases in cheapening the cost of production. However, dew retting is not often carried on in large enough volume to warrant the using of machines, the work being practically all done by hand. Other factors that affect the quality of fiber in water retting are important in dew retting and affect the quality somewhat similarly.

Atmospheric conditions here in Oregon are not well suited for dew retting, largely because of the cool evenings and warm, clear days in addition to a lack of moisture in the form of dews and rains, for the most part, during the summer months. Too much

moisture during the spring and fall months during most years makes this form of retting quite unreliable. These undesirable or unfavorable conditions for dew retting favor water retting.

The organisms responsible for dew retting have not been studied extensively, but it is believed that they are largely aerobic in nature. The work of Behrens (10) reported in 1903 has formed the basis for subsequent investigations. His findings have been verified and reported on by Ruschmann (67) who states that Mucor plumbeus and Cladosporium herbarum are the active agents, the latter being more widely distributed and somewhat more active than the former. Rhizopus nigricans may be present but has very little effect, especially in hemp retting.

Also, Muller (34) includes the giant mold Mucor stolonifer genannt and Behren's Mucor hiemalis which rets at a low temperature, 2-5° C., the optimum being less than 30° C.

In summing up dew retting out of doors Ruschmann (71) states the uncertain issue of the ret, the dependence on weather, and the fact that good quality fiber is seldom obtained, as well as diminished yield of fiber, are all against using the best quality of flax straw for dew retting. The cost of dew retting is less than with warm water tank retting as is borne out by Tobler (82) who states that from 50-100 per cent more labor is required in the factory retting of flax by the common European processes than is necessary in dew retting as practiced by farmers.

Attempts have been made to overcome the major difficulties of the natural dew retting process by placing the flax straw under artificially controlled conditions. Not only the retting bed but the atmospheric conditions would be controlled so a better quality of fiber should be produced.

A process was developed by Ochmann (German patent 8-P.340, 412), (66), in which the flax straw was packed in tanks and water allowed to fall upon it in the form of a heavy rain for about two hours every other day. The same water was used time after time, being subjected to a simple oxidizing treatment and the removal of a large proportion of the impurities by allowing them to settle out between each sprinkling. About three weeks time was required to complete the retting process.

Cromer's process (U.S. patent No. 1,448,391, March 13, 1923) consisted of stacking the flax, hemp, perini or jute stalks in superimposed layers in an inclined position when harvested. After curing, moisture was added so as to facilitate retting, after which the stack was aerated.

De Geyter (Belgium patent No. 364,851, Nov. 30, 1929) employs a combination of water retting followed by dew retting. The textile material is immersed in a solution consisting preferably of diluted liquor in which flax has been macerated to which are added nutritive substances and spornulated cultures of active *Mucor*. The material is warmed for one to two days and then spread out in the open or in rooms at 30° C. in which the air is kept moist.

It is very evident that the above process is expensive, even more so than warm water tank retting. More handling of the straw is required in addition to more elaborate equipment and nutritive materials and cultures. All of these disadvantages plus the longer time necessary to complete the process without any improvement in quality of fiber makes this process along with a number of others quite impractical.

In summing up biological retting of fiber flax, the percolation system of warm water tank retting predominates over all other methods or practices used during the past and at the present time. River retting is being used somewhat in Europe. It is really a very rapid form of percolation or channel retting as regards the volume of water passing through the retting material. Channel retting in tanks has been unsatisfactory. The addition of salts and pure cultures of organisms to the solution has aided somewhat in hastening the retting process but without any improvement in quality of fiber. The extra expense and handling of these materials have not cheapened the cost of production enough by shortening the retting period to warrant their adoption. Dew retting gives variable results and requires too much time and space under natural conditions. Under controlled conditions more satisfactory results are usually obtained but no reduction in cost and a longer period of time is required to complete the retting process.

CHEMICAL RETTING

The using of chemicals to ret flax has been attempted for over a century and a half to a limited extent. It has only been during the past two decades, however, that the demand for some quick method of loosening the fibers has become imperative if large scale operation is to be applied to the production of linen. During this time many processes have been developed and numerous patents awarded. Many of these processes consist largely of promotional schemes in which the main object has been to sell stock in some company rather than to explain the reactions that occur, or really to contribute something to the sciences of flax retting. Also, most of the processes developed have been the result of research with small quantities of material in the laboratory and a theoretical transformation is made to hypothetical commercial scale operation. Numerous patents have been obtained for these processes without them being given a critical test by the inventors in order to make known to industry the usefulness or uselessness of the process.

Literature Review

Of the early chemical processes which were used to any extent in the industrial maceration of flax, Bazzocchi (9) mentions those of Professor Pietre Willermoz di Liene and of Abbot Rezier in 1786 in which organic acids and sulphur were the active reagents. Then, in 1789 an unknown member of the Patriotic Society of Milan claimed

that hemp could be retted with vinegar and the yolk of egg. He also mentions that Thummler, Leidel and Baur used alkalies and acids, while Nicelle and Schmidt of London, De la Roche of Paris, and Sampson of Glenalmond proposed methods based on the use of acid salts of soda and soap.

With the beginning of this century many processes were developed, most of which were never used on a commercial scale. In 1902 the Cheveline Process (5) was used in a mill near Moscow, Russia. The process consisted of treating the flax with alcohol, then with mineral oil and steam. It was claimed that linen cloth could be produced for $13\frac{1}{2}$ cents per yard. A little later the Rosseau Process (4) was developed in which the straw was bleached, previous to being macerated in a chemical solution composed of sodium carbonate, sodium sulphate and soap containing petroleum ether. The Roger's Process (3) consisted in boiling the flax straw in an emulsion of linseed oil for an hour. The resulting fiber was weak and quite uneven and, therefore, not suited for most purposes.

Bradshaw (12) developed a process which consisted of alternate alkali and acid treatments, the principle of the process being based on the generating of carbon dioxide gas by the action of an acid on the impregnated soda solution, the expansive force of the gas splitting up the fiber into ribbon-like filaments resembling cotton.

Milk casein was used in combination with sal soda to ret flax by an unnamed chemical process. It was claimed that greater strength

was retained with improved nature and dyeing properties (6).

Hydrocarbons have been used as retting reagents to some extent. The Douglas Process (U.S. Patent No. 1,224,722, 1917) was based on the use of kerosene and gasoline. The fiber produced was quite unsuitable for spinning purposes, being coarse and harsh as judged by McColl (50). Another process developed a little later and which gained widespread publicity, being tested probably more widely than any other chemical process, was that of Peufaillit. The process consisted in using about 4 per cent petroleum oils under pressure in autoclaves. A somewhat extensive description of the process is given by Ruschmann (68). He criticizes the process by stating that equally good results are obtained by using steam alone as with the petroleum. He considers that the materials leached out of the stems into the cooking liquor, largely acids, bring about the hydrolysis of pectins and weaken the fiber. Results in general have not been satisfactory and the process has been discontinued almost entirely.

Many processes developed have remained secret or at least some secret chemical solution was used to effect the loosening of the fibers from the flax stems. An example of this type of process is that of Robinson (British Patent No. 141,982, Apr. 23, 1920), which probably received the largest amount of financial support and which turned out to be nothing but a stock selling proposition. The process itself involved the using of decorticated fiber, retting being accomplished by the use of several chemical solutions including a vegetable oil. Extensive handling of the retting material was

necessary which, in addition to the cost of chemicals, made the process too expensive to ever be adopted. The same criticism holds for numerous other processes that have been developed.

The retting property of soaps has been known for over a century, but it has been only since the beginning of this century that any appreciable amount of work has been done with these materials. Pritchard (57) mentions the inventions of Lee back in 1812 and of V. P. G. De Moor¹ in 1855 in which soaps were used to ret flax.

About three decades ago Pritchard began investigations on soap retting and since that time has probably done more work with soaps than any other investigator. By 1909 he had developed a retting process whereby a fairly good quality of fiber was produced. Decorticated fiber was treated in a 4 per cent soap solution for 5 hours at 100° F. (56). It was not until 1927, however, that his process was used on a commercial scale. The Pritchard Flax Fibre and Pulp Company was organized and two plants built, one at Bushby, near Glasgow, and the other one at Dromora, near Belfast. The undertaking was a failure and the company was dissolved in 1929.

Kidger and Harris (British patent No. 190,198, May 15, 1922) used soap to degum various textile fibers. 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, gasoline, or the like

¹

De Moor, V. P. G. *Traitee la culture dulin*, p. 17, 1855.

was added and boiling continued until digestion was completed. This process, however, failed to become popular and has never been used to any extent.

The Maid-O'-Kelp Co., Inc., Seattle, Washington, attempted to ret flax straw with a kelp soap which they produce. The advantage claimed by their process is that their soap, which is high in iodine, would bleach the fiber thereby making it unnecessary to rebleach when in the form of twisted rove as is usually necessary with ordinary water retted fiber. The results of their trials at the Salem Linen Mills, Salem, Oregon, were not very satisfactory because the retted fiber, aside from lacking uniformity in color, retained so much soap that it could not be spun (60).

Robinson and the writer (62) retted small lots of flax straw with a number of pure soaps and kelp soap. Most of the samples retained an appreciable quantity of soapy material. Kelp and stearate soaps gave a more bleached fiber with a smaller quantity of adhering soapy materials than the oleate soaps. Also, the fiber strength tests of the stearate and kelp soap retted flax straw were almost equal to those of fiber from natural water retted straw. Laboratory tests were conducted on small samples of decorticated fiber. Results similar to those reported above were obtained for the different soaps.

Lowry and Grant (11) found soap retted decorticated fiber difficult to scutch because of a matting of the fibers in drying. This gave a low hackling percentage. They found it difficult to

remove all of the soapy materials by washing. A very fine fiber was obtained, however, which was spun to 160 lea.

Various materials in addition to those previously mentioned have been used in attempts to ret flax. Some of the more important ones include: sodium carbonate, sodium oxalate, ammonium oxalate, ammonium sulphate, ammonium chloride, sodium phosphate, sodium hydroxide, and various derivatives of hydrocarbons including ethelene dichloride, ethelene tetrachloride, ethelene pentachloride, etc. Also, combinations of some of these materials have been used.

Of these materials listed above, the sodium and ammonium oxalate salts were the only ones that have been at all effective in loosening the fibers in flax stems.

Muller (52) states that the Oberfeur Hamm Process developed in 1931 shows greater possibilities than any other chemical method for the retting of flax. He states that the cost of preparation is lower and the time necessary to complete the process is shorter in contrast to the customary practices in use. Several lots of flax straw were run on a commercial scale at Sorau, Germany, with favorable results. However, a sample of flax straw sent over there from the State Flax Industry, Salem, Oregon, produced fiber quite unsuitable for spinning purposes. The process has not been used commercially to date, which may be due to unforeseen complications or lack of financial support.

During the past two years several new chemical retting processes have been described, among which are the Geiss (31) and

the Semmes (74) processes. It is claimed by the former that a yield of 16 per cent fiber is obtained at a cost that makes it possible for linen to compete with cotton selling for 10 cents per pound in New York City. Both of the above processes utilize secret degumming solutions.

In summing up the literature on chemical retting numerous processes have been developed but not one of these processes has brought about any improvement in quality of fiber or lessened cost of production as compared to natural water retting.

MATERIALS AND METHODS

Chemical

The first part of the work consisted of retting small samples of flax stems in six-inch soft glass test tubes using a rather wide variety of chemicals in an attempt to find some that would effectively loosen the fiber bundles from the woody portions of the flax stem. Small sections of flax stems were used in this work because it was possible to determine by the loose core test described by Davis (21) whether a particular chemical solution loosened the fibers. This did not necessitate the drying and subsequent working up of samples that were not retted when removed from solution. A sharp razor blade was used to cut the sections of flax stems so as not to tear or crush the ends of the stems. In all cases the test tubes were completely filled with stems so that the proportion of

solution to straw would be kept as low as possible. Chemically pure materials were used, the concentrations being determined accurately by weighing, pipetting, or titrating.

Various concentrations of chemicals were used for varying lengths of time at room temperature, steam oven at 70° C. and on the steam bath. Corks were used to keep the stems completely submerged in the chemical solution and to aid in preventing evaporation. The test tubes were washed thoroughly before being used each time to prevent any contamination from a previous retting. Immediately upon removal from the retting solution several stems were tested by the loose-core method previously mentioned. Those samples which were judged to be insufficiently retted were either given a more extended treatment or were discarded without further analyses. The samples that were retted sufficiently were removed from the retting solution and washed thoroughly, first in warm and then cold water, before being placed upon wrapping paper to dry.

The later work consisted of degumming decorticated fiber. Short sections of flax stems were decorticated by passing them through a small hand brake to remove approximately one-half of the woody material. Eight-inch soft glass test tubes served as containers for most of the work, 200 cc. beakers being employed in a few of the tests. All of the degumming was done on the steam bath as unsatisfactory results were obtained at room temperature, and, for the most part, in a steam oven at 70° C. in the first part of the work.

A supply of chemically pure soap and ammonium oxalate solution was made up previous to the running of any experiments. Potassium oleate was selected as the soap to be used, as no apparent differences showed up in the first part of the work between the various soaps. Sodium and ammonium base soaps were used in a few trials previous to the using of the potassium base soap. In making up the soaps, the fatty acid was dissolved in alcohol and warmed before the base was added. This was found to produce a better quality of soap than when the alkali was added directly to the fatty acid.

In some of the trials various proportions of soap and ammonium oxalate were mixed together in an attempt to produce a fiber mid-way between that produced by either of the chemicals alone. Also, attempts were made to ret decorticated fiber with solutions recovered from previous retting tests. Usually there was just enough solution remaining from two duplicate tests to be used in one subsequent test. All the degummed samples were washed, previous to being dried outside. Some samples were washed with very dilute acids and alkalies in an attempt to remove the adhering soapy materials which are difficult to remove by ordinary washing. Also, subsequent soaking in a warm solution of ammonium oxalate was tried.

A small, specially patented hand brake with two fluted rolls was used to remove the woody material from the fiber. A small hackle was used to comb out any remaining shives and short fibers.

No weights were taken as the prime interest was in the quality of fiber that could be produced. Strength tests were run on some of the degummed fiber samples by conditioning small samples of fiber 10 cm. long at 65 per cent humidity previous to weighing and breaking. A Scott fiber strength testing machine was used to determine accurately the breaking strength of the samples.

The quality of the fiber was determined largely by the feel when gently pressed and rubbed between the thumb and forefinger. Also the color, fineness and strength were considered, the latter being roughly tested by breaking several strands, the force required to break the strand being a rough approximation of the strength of the sample.

Biological

Four small retting tanks 4 x 1 x 1 feet were constructed out of one inch fir boards, the corners being coated with putty and white lead paint to aid in water-proofing them. Two of the tanks were equipped with 30 feet each of soil heating cable and a thermostat to maintain a constant temperature of 80-85° F. This was considered "optimum" for natural warm water basin retting and was used in all the retting tests.

A large supply of uniform quality straw was used as a source of material for all of the tests conducted. The straw averaged about 40 inches in length, being of average size in diameter, light colored, clean, and was harvested at late yellow maturity. The straw was threshed with a regular flax thresher by passing the heads

between rapidly revolving rollers, later being stored in a shed until ready for use.

In preparing samples for retting tests, all short fine stems were removed and the bundles were squared-up so as to present a very neat appearance before being weighed on a gram balance.

In the case of water retting, the water was allowed to heat up to 80-85° F. before the samples were put to ret. The completion of the ret was determined by the loose core test previously mentioned. Upon removal of the samples from the retting liquor, they were washed thoroughly with cold water before being set up in stocks in the greenhouse to dry.

Several samples of straw after being weighed were decorticated by means of a small hand brake. The loss in weight due to decortication varied from 10 to 40 per cent with different samples. The former consisted mainly in crushing the stems and breaking off of the roots.

This was performed to find out if possible whether breaking of the unretted flax straw weakened the fibers as has been stated by some workers. Also to find out whether the retting process would be hastened due to the greater ease with which the organisms could reach the pectin substances. The retting process was allowed to continue 7 days.

Ordinary tap water was used for all of the tests except one in which old retting liquor was filtered through a 4-inch layer of medium fine sand. The filtered solution retained its dark color

and a large portion of its sediment which was allowed to settle, the upper portion of the liquor later being siphoned off into another retting tank. No fresh water was added to the old filtered liquor. The samples of flax straw were removed at the end of 10 days even though they were not sufficiently retted.

The retted samples were broken or braked by the aid of a small hand operated brake, the shives removed by intermittent shaking and rerunning of the fiber through the brake as previously described.

To carry on the dew retting tests, the two tanks equipped with soil heating cable and thermostat were covered with two layers of heavy tar paper so as to keep a constant temperature of 80-85° C. A modification of the Ochmann process previously described was employed, moisture being supplied by spraying water from an adjoining retting tank which served as a reservoir, at room temperature on the flax straw by the aid of a Brown's No. 9 hand sprayer for 5 minute periods daily. Also a quantity of water remained in the bottom of the tanks so as to aid in maintaining a very high humidity within the closed retting chambers.

During the first tests the tanks were allowed to remain upright as in the water retting. The samples were spread out more or less uniformly over the bottom of the tanks, being supported by small laths. Later, small bundles were placed in the tanks without being spread out. One tank was inoculated with leaf mold to increase the growth of molds over the surface of the flax straw.

In the final dew retting tests the tanks were set on end so as to spray the water down the stems from above which was thought would give a more uniform distribution of the moisture over the entire surface of all of the flax stems, both inside and on the outside of the bundles. Daily watering was followed as in the previous tests. It was found more difficult to keep as high a humidity with the tanks in this vertical position due to greater evaporation. No weights were taken of the straw as the principal interest was in quality of fiber which would have later been followed by quantitative tests if the preliminary ones had given satisfactory results.

Experimental Data and Discussion

The effect on loosening of the fiber from small sections of flax stems using a wide variety of chemical solutions is tabulated in table 1.

Of the chemicals employed to ret flax, soaps and oxalate salts gave the most satisfactory results, the former being somewhat better than the latter. All of the acids produced a somewhat similar effect in that they effectively loosened the fibers when placed in the steam bath for a period of time but upon drying the fibers became more or less securely fastened back onto the remainder of the central woody core of the flax stem. A partial reason why it was difficult to remove the fiber upon drying was that the central portions of the flax stems and probably a portion of the softer

Table 1. Results on loosening of flax fibers obtained with small sections of flax stems by using various concentrations of chemicals at various temperatures, for varying lengths of time.

Chemical	Concentration	Time	Temperature	Condition of fiber	
				when removed from retting soln.	when dry
		hours			
Acids:					
Sulfuric	0.1-3.92N	1-24	room at 20°C.	not loose	
		$\frac{1}{2}$ -24	oven at 70°C.	not loose	
		$\frac{1}{2}$ -5	steam bath	loose	not loose
Hydrochloric	0.1-4.5 N	1-129	room at 20°C.	not loose	
		1-24	oven at 70°C.	slightly loose	not loose
		1-5	steam bath	loose	not loose
Acetic	0.1-2 N	1-120	room at 20°C.	not loose	
		1-5	steam bath	loose	not loose
Citric	0.1-1N	1-5 $\frac{1}{2}$	steam bath	med. loose	not loose
Oxalic	0.1-1 N	1-2	steam bath	loose	not loose
Above acids followed by washing with dilute alkali					not loose
Bases:					
Sodium hydroxide	0.1-1 N	1-5	steam bath	loose	not loose
		1-24	oven at 70°C.	sl. loose	not loose
		1-46	room at 20°C.	not loose	
followed by acetic, citric & sulfuric acid baths		1-5	steam bath	loose	not loose

Table 1 cont'd.

Ammonium hydroxide	0.1-1 N	1-5	steam bath	loose	not loose
		1-46	room temp. at 20°C.	not loose	
Calcium hydroxide	0.5-1 %	1-5	steam bath	not loose	
" -- sulfuric acid	0.5-1%&0.1N	3&1	steam bath	med. loose	not loose
Potassium hydroxide	0.5-1 %	1-5	steam bath	loose	not loose
Salts:					
Sodium carbonate	0.83-16.6 %	1-47	room temp. at 20°C.	not loose	
		1-5	steam bath	med. loose	not loose
Sodium meta silicate	0.25-4 %	1-100	room temp. at 20°C.	not loose	
		1-5	oven at 70°C.	not loose	
" " " -- soap		1-30	room at 20°C.	not loose	
		1-30	oven at 70°C.	sl. loose	not loose
		1-7	steam bath	loose	med. loose
Saponin	0.5-1 %	1-5	steam bath	not loose	
" -- soap	0.5-1 %	1-5	Steam bath	loose	loose
" -- acetic acid	0.8&0.2%	1-5	steam bath	not loose	
" -- sodium meta silicate	0.8&0.2%	1-5	steam bath	not loose	
Blood albumen	1.0 %	1-5	steam bath	not loose	
Glycerine	10.0 %	1-5	steam bath	not loose	
" -- soap	10.0 & 2.0%	1-5	steam bath	loose	loose
Ammonium oxalate	0.5-Sat.	1-5	steam bath	very loose	loose
Sodium oxalate	0.5-5.0 %	1-5	steam bath	very loose	loose

Table 1 cont'd.

Soaps:

Sodium palmitate	0.1-3.0 %	1-5	steam bath	very loose	loose
Sodium oleate	0.1-3.0 %	1-5	steam bath	very loose	loose
Sodium stearate	0.1-3.0 %	1-5	steam bath	very loose	loose
Potassium oleate	0.1-3.0 %	1-5	steam bath	very loose	loose
Ammonium oleate	0.1-3.0 %	1-5	steam bath	very loose	loose

tissues surrounding the fiber bundles were dissolved thereby leaving the central woody cores weak and flexible rather than brittle as is the case in natural water retted straw.

With the more concentrated acid solutions a serious weakening of the fiber resulted which was due probably to a weakening of the junctions between the ultimate fiber cells and the harsh mechanical treatment necessary to remove the fiber. The fiber was coarse and had a harsh feel which qualities are very undesirable for spinning purposes.

Alkalies had a very unfavorable effect both when used alone and when following an acid treatment. With the more concentrated solutions the macerated stems took on a reddish cast, the degree of redness varying with the concentration. When dried the straw appeared as a reddish, snarled mass, the cortex of the stems being mostly dissolved and rubbery, which made it impossible to remove the fiber. With more dilute solutions the effects were less drastic, but it was difficult to remove the fibers because of the fragile and rubbery cortex. Gillis (British patent No. 297,302, February 15, 1927) used potassium chromate as a hardening agent which facilitated the removal of the fiber. Sodium hydroxide had a more drastic effect than either the ammonium or potassium hydroxides. Calcium hydroxide was ineffective as a macerating agent which is explained by Zakochtchikov et al (90) who states that calcium and magnesium compounds form insoluble salts of pectic acid, while sodium and ammonium compounds form soluble salt with

anhydroarabinogalactosemethoxytetragalacturonic acid, which constitutes pectin and can be readily removed from the flax stems. They claim that this also explains the specific action of sodium and ammonium oxalate salts which have long been used as pectic solvents and which have given such good results in the maceration of flax. Calcium oxalate is precipitated out of solution as the result of a double decomposition.

Koryheniovskii (40) in comparing sodium carbonate and sodium oxalate showed that the latter does not effect as complete a purification of the fibers as the former but that the loss in weight was greater with the sodium carbonate.

From the results presented in table 1, sodium and ammonium oxalate were much more effective in loosening the fibers than sodium carbonate which seemed to give a more brittle central woody core. The fiber from both the oxalate and carbonate treatments was rather coarse and dry, the former giving a better bleached fiber which is quite desirable.

Pure soaps of sodium and ammonium palmitate, oleate and stearate gave quite satisfactory results, no particular one being outstanding.

Robinson and Johnston (62) found stearate soaps to give better results than oleate soaps, especially as regards ease in removal of soapy materials that remain on soap retted fiber. Also, a stronger and better bleached fiber was produced by the stearate soaps.

The satisfactory results obtained by using soaps led to a more intensive study on the use of soaps as maceration agents with decorticated fiber. Also, ammonium oxalate was included and the results of this work are reported in table 2.

Degumming of decorticated fiber by soaps gave fairly satisfactory results when used three hours or more on the steam bath at or near 100° C. It was found difficult, however, to remove all of the adhering soapy materials from the fiber. Warm, running water for a period of time was effective, but the amount of water required to completely remove these materials would be prohibitive from an economic standpoint. Cold water was quite ineffective even if allowed to flow through the fiber for a long period of time. With these unsatisfactory results, acids and alkalies were used. Dilute hydrochloric and acetic acids quickly removed the soapy feel of the fiber but upon drying the fiber was slightly sticky, indicating perhaps that small amounts of fatty acid or some fat-like material remained on the fiber. With acetic acid in addition to this sticky feel, the fiber retained an odor resembling vinegar which was quite disagreeable.

Washing with dilute sodium hydroxide was very unsatisfactory, the fiber being more sticky than when washed in a small amount of cold water. Ammonium hydroxide acted similarly to acetic acid, except that the fiber retained an odor of ammonia.

The degummed fiber with both the soap and ammonium oxalate treatments was partly bleached, the latter being more effective than

Table 2. Results of treating decorticated flax fiber with various degumming agents on the steam bath for varying lengths of time.

Degumming agent	Concentration	Length of treatment	Subsequent treatment	Remarks
	per cent	hours		feel of fiber
Potassium oleate	1.0	3	washing in warm water	soft, fine fiber; free from soap
"	"	"	" " cold water	not entirely free from soap
"	"	"	" " both cold and warm water	same as with warm water
"	"	"	washing in dilute hydrochloric acid	slight sticky feel
"	"	"	same with acetic acid	" " "
"	"	"	same with ammonium oxalate	same as with warm water
"	"	1.0	4 & 5	same treatments as above similar results
"	"	1.0	1-2 $\frac{1}{2}$	" " " " not sufficiently retted
"	"	1.0	1	washed in warm water then treated 1 hr. with 0.5 % ammonium oxalate on the steam bath
"	"	1		slightly more coarse and dry; loose
2/3 " " plus 1/3 ammonium oxalate	1.0 & 0.5	1-3	washing in warm water	not entirely loose; slightly harsh and dry
1/2 Potassium oleate & 1/2 Ammonium oxalate	1.0 & 0.5	1-3	" " " "	slightly more dry and harsh

Table 2 cont'd.

1/3 Potassium oleate & 2/3 Ammonium oxalate	1.0 & 0.5	1-3	washing in warm water	more harsh and slightly more coarse
Sodium oleate	1.0	$\frac{1}{2}$ -1	" " " "	not sufficiently retted
Ammonium oleate	1.0	$\frac{1}{2}$ -1	" " " "	" " "
	3.0	$\frac{1}{2}$ -1	" " " "	fibers fine and loose
Used Potassium oleate*		2-5	" " " "	not sufficiently retted
" " " & " Ammonium oxalate**		2-5	" " " "	variable, some fiber good
Ammonium oxalate	0.5	1-5	" " " "	fibers loose, dry, coarse, some weak and brittle
Used Ammonium oxalate*		2-5	" " " "	better quality than from first treatment
Ammonium oxalate	0.5	1-3	" " " " & treated with 1.0% Potassium oleate on steam bath 1 hr.	fibers loose, fine and of fairly good quality; low yield of fiber

* Old liquor from previous degumming solutions used from 2-5 hours in the previous treatments.

** Same as above except from various combinations or proportions of Potassium oleate and ammonium oxalate solutions used from 1-3 hours in the previous treatments.

the former. This is an advantage in favor of chemical retting as most fiber that is used for linen thread must be bleached. The quality of fiber and the degree of bleach desired affect the loss in weight due to bleaching but this loss may run up as high as 25 per cent or even more in extreme cases. In addition to this loss in weight and probably a similar loss in strength, the process is expensive, requiring extra handling, chemicals, washing, drying, etc. Bleaching would not always be so necessary with fiber degummed by these solutions. Usually, however, only a partial bleach is obtained with the soap and if a complete bleach were desired, it would be necessary to complete the bleaching later on before the thread could be made into cloth. This would involve the same amount of handling but a shorter period in the bleaching solution.

Ammonium oxalate produced a rather coarse, dry, harsh feeling fiber which seemed to be brittle, especially when treated for more than two hours in the steam bath. No difficulty was encountered in removing the oxalate from the degummed fiber. Solutions previously used to degum samples of fiber gave quite satisfactory results. The fiber was less harsh, finer and seemed to be much stronger. It had a bright luster and was pure white in color. Very little difference was apparent between the samples degummed in solutions that had been used one, two, three or four hours in the previous treatment.

The explanation of this good quality of fiber is difficult and probably not a dilution factor as more dilute solutions than

0.5 per cent were unsatisfactory. There is a possibility that due to some previous chemical combination of the ammonium oxalate with pectic materials and other organic salts in the decorticated fiber, a combination of physically active substances was produced which aided in the loosening of the fibers or a breaking down of the binding materials. At any rate, an equally effective loosening of the fibers resulted and a better quality of fiber was produced.

Similar results were not found to occur when soaps were used the second time. The effect of soaps in the loosening or degumming of fiber has not been studied to any extent. Whether or not it is a chemical reaction is not definitely known, but it seems probable that it is more of a physical reaction, in that soaps are quite stable and not readily broken down. Some have thought it to be a wetting effect as soaps are good wetting agents, but based upon our experiments this is not believed to be the case. Saponin, also a wetting agent, failed to loosen the fibers satisfactorily.

Because of the fact that soap solutions fail to degum more than a certain quantity of fiber would suggest that the soap is altered in some way so that it is no longer effective. Also, the fact that very little difference is apparent between samples treated for three, four or five hours as regards quality and strength would support the above suggestion. How long it would be possible to leave fiber in the degumming bath without deterioration in quality and strength is not known as the mere physical action of heat in the presence of moisture has a tendency to break

down the binding materials of the fiber bundles and would, no doubt, cause a serious weakening of the fibers after a period of time. Further investigations along this line would be of interest.

No significant difference in strength is apparent between the soap and ammonium oxalate retted fiber as is shown in table 3. In the case of the latter, not only did the two-hour treatment give a stronger fiber but also a cleaner fiber than the one-hour treatment. This is probably due to the fact that the fiber in the latter case was not sufficiently retted and because of the severe mechanical treatment necessary to remove the shives, the fiber was materially weakened. When treated for three hours or more it would appear that a slight weakening of the fibers resulted as severe mechanical treatment was not necessary to remove the shives. This supports the view that the ammonium oxalate enters into chemical combination with the pectic materials and with an excess of chemical, the reaction continues to the extent that the pectin, which binds the ultimate fibers, is attacked, thereby weakening the fibers.

All of the fiber strength tests were low as compared to those of fiber from natural water retted straw. However, the chemically retted fiber averaged almost as strong as the water retted decorticated fiber. Robinson and Johnston (62) found the strength tests of soap retted flax straw to vary somewhat for the different soaps but that they were practically equal to those of fiber from natural water retted straw. These results show quite conclusively that the fibers were weakened by decortication of the unretted flax stems.

Table 3. Comparison of strength of flax fiber degummed with soap and ammonium oxalate solutions on the steam bath for varying lengths of time with fiber from natural warm water retted flax straw.

Degumming solution	Concentration	Length of treatment	Average* weight of sample	Average breaking strength per gm. of fiber	
				kgs.	kgs.
Potassium oleate	1.0	2	0.0437	6.64	151.5
		3	0.0439	5.90	134.4
		4	0.0376	6.42	170.8
		5	0.0543	9.46	174.2
Ammonium oxalate	0.5	1	0.0569	7.68	135.0
		2	0.0629	12.50	198.7
		3	0.0352	5.00	142.0
		4	0.0347	4.58	132.0
		5	0.0325	4.27	131.4
Potassium oleate followed by Ammonium oxalate	1.0 & 0.5	1 each	0.0636	11.88	186.8
Natural water retted straw		7 days	0.0402	18.66	464.2

* Average of 10 samples of fiber selected at random from the larger samples.

Water Retting Experiments

Fiber of high quality was produced by retting in the usual manner with warm tap water. The fiber was uniform, medium fine and very strong in addition to having a bright luster. In contrast to this fiber, that produced from straw retted in old retting liquor was of very low quality and could not be freed entirely from its shives. A comparison of the retting losses, scutched fiber and fiber strength tests is shown in tables 4 and 5. Apparently, the old retting liquor acted unfavorably towards the developing flora of the new straw and, as a result, very little loosening of the fibers was effected. The loss in weight due to retting was the same as with the fresh water which would indicate that some change other than the leaching out of the soluble materials had taken place. It seems probable that cellulose rather than pectin consuming organisms functioned as when straw is allowed to remain in the retting solution too long it is this flora that develops and a serious weakening of the fiber results. This is borne out by comparing the strength tests shown in tables 4 and 5.

In addition to producing a weak fiber that could not be freed entirely of its shives, the fiber was of a dark brown color, coarse and had a harsh feel. Better results, no doubt, would have been gotten by using only a small portion of the old retting liquor, especially if some alkaline substance had been added to neutralize the harmful acids present. As was mentioned previously, this was

Table 4. Showing limited variation in loss from retting, scutching and hackling of 4 uniformly treated samples of natural warm water retted flax straw.

Sample No.	Weight deseeded straw	Weight retted straw	Loss in retting	Scutched fiber in per cent weight deseeded straw		Hackled fiber in per cent weight deseeded straw	
	gms.	gms.	%	gms.		gms.	
1	1500	1190	20.67	328	21.87	187	12.47
2	1500	1197	20.20	341	22.73	193	12.87
3	1500	1205	19.67	354	23.60	195	13.00
4	1500	1200	20.00	352	23.47	229	15.27
Average	1500	1198	20.27	344	22.92	201	13.40

Table 5. Effect of retting deseeded flax straw in old retting liquor and in fresh water upon retting losses, per cent of scutched fiber and fiber strength.

5

Nature of ret	Length of retting period	Weight deseeded straw	Loss in retting	Scutched fiber		Fiber strength tests		
				weight	in per cent deseeded straw	Average weight sample	Average	breaking str. per gm. fiber
	days	gms.	%	gms.		gms.	kgs.	kgs.
In old liquor	10	560	19.1	152*	27.14	0.0430	7.35	170.9
In old liquor	10	560	19.1	143*	25.54	0.0485	8.10	164.9
In fresh tap water	7	1500	20.3	344	22.90	0.0402	18.66	464.2

* Fiber was not entirely free from shives.

the principle of the Vansteenkiste, Coussine and other processes which are used to a limited extent.

The results obtained from retting decorticated fiber were far from satisfactory. First, it was almost impossible to determine when to terminate the ret as the loose core test could not be applied. It seemed to be more of a guess as no standardized method has been evolved for this purpose. Second, after the decorticated fiber was retted and washed thoroughly, it had to be placed on some clean structure, or hung up to dry as it could not be set up in stooks like the bundles of straw. This was quite inconvenient except when dried artificially. Third, upon drying the fibers seemed to become more or less matted with the result that the shives were difficult to remove.

This is in harmony with the findings of Kraus (41) in which he reports that previous breaking of the stems appears to favor a quicker ret, but that it is considered much more advantageous to steep the stems whole, as the woody portions can then be removed intact, whereas with broken stems particles of wood become entangled among the fibers.

The yield of scutched fiber was equal to that from the retted straw but was darker in color, more coarse and had a more harsh feel. In addition, the decorticated fiber was much weaker as is shown in table 6, which would indicate that the fiber was weakened by being broken before retting. Semmes (74) claims that this has been responsible for the failure of degumming processes in the

Table 6. Effect of retting lots of decorticated flax fiber in water at 80-85°F. for varying lengths of time.

Sample No.	Length retting period	Weight deseeded straw	Weight decortic'd fiber	Loss due to breaking	Weight retted fiber	Loss in retting	Scutched fiber weight	Scutched fiber in per cent deseeded straw	Hackled fiber in % wt. deseeded straw	Breaking strength/ gm. of fiber
	days	gms.	gms.	%	gms.	%	gms.		gms.	kgs.
1	7	200	120	40.0	112	6.67	44	22.0	13.8 6.90	187.6
2	7	200	135	32.5	123	9.00	46	23.0	14.6 7.30	137.7
3	7	200	180	10.0	151	16.11	47	23.5	14.9 7.45	189.5
4	5	100	50	50.0	47	6.00	22.5	22.5	6.30 6.30	147.7
5	5	100	80	20.0	68	15.00	23.4	23.4	5.10 5.10	130.5
6	3	100	80	20.0	69	13.75	24.6	24.6	5.25 5.25	73.6
*	7	1500			1198	20.27	344	22.9	201 13.40	464.2

* Natural water retted straw.

past. He claims recently to have perfected a machine that will not stretch or break the unretted fibers which may have a marked effect on improving the degumming of fiber biologically as well as chemically.

The degree to which the flax straw was decorticated did not seem to show any consistent differences in strength of the fiber. Sample number 6 which was only partially retted was very difficult to clean up and the fibers were probably weakened by the mechanical treatment necessary to remove all of the shives. Also, the fibers in addition to being coarse and harsh were partially coated with gummy residues that could not be removed by scutching and hackling. These adhering materials increased the weight of the fiber strength samples over what they would have been provided they had been removed in the retting process and thereby lowered the breaking strength per unit weight of fiber. This may partially explain the lack of uniformity in the results presented in table 6.

The saving of a day or two out of six or seven for the ordinary tank retting method probably is not enough to warrant the extra handling of the straw to decorticate it previous to retting. This would be especially so if retting was delayed until the following summer as is the usual practice when time is not as important a factor as at harvest time.

In summarizing retting of decorticated fiber, the advantages include: 1. a slightly shorter retting period, 2. less bulk to handle and 3. a smaller space or volume of solution is necessary per

unit of fiber for retting. The disadvantages include: 1. an extra handling of the straw, 2. more mechanical treatment, 3. more special care in drying which, if artificially dried, increases the cost, and 4. a poorer quality of fiber. It is apparent that the retting of decorticated fiber is more expensive because of the extra handling and care of the fiber in comparison to the retting of straw. Therefore, because of the greater expense and poorer quality of fiber that is obtained, the retting of decorticated fiber is not warranted by methods available at the present time. If some method can be devised so as not to injure the fibers upon decortication, a more thorough consideration of this form of retting will be in order.

Dew Retting Experiments

The dew retting process somewhat approached that of Ochmann's being modified to the extent that water was applied every day for five minutes instead of every second day for two hours. Weights of the deseeded straw were taken in all cases but due to the poor quality of fiber produced no further data were considered worth while.

In the first trials a rather poor growth of molds developed over the surface of the retting flax straw. It was noticed by using the loose core test that the lower portions of the flax stems retted much more slowly than the middle and top portions of the stems. Sellegren (73) states that this is due to the presence of larger quantities of gluten in the lower portion of the stem and in the root which is slow to dissolve. The top ends of prepared

flax fiber have a darker color and less strength than the root ends. This phenomena also occurs to a less marked extent in water retted straw. In general, however, this condition is overlooked as it probably could not be overcome and would not be practical.

The stems on the immediate surface seemed to ret more rapidly than those just below. Also, these stems retained a better color than those in contact with the laths on the bottom of the retting tanks. There was not much difference in quality of fiber from straw that retted 12 and 18 days.

It was quite coarse, harsh and had a dark brown color. The fiber had numerous dark spots which were more or less rotten, thereby weakening the fiber greatly. This probably was due to colonies of cellulose decomposing organisms developing at these points.

A luxuriant growth of molds developed on the small bundles that were inoculated with leaf mold. After retting for two weeks the straw on top was fairly well retted but on the inside and on the lower side of the bundles the stems had not started to ret. A gelatinous-like layer of gummy material formed a water-proof layer over the entire top surface of the flax bundles, thereby serving as a watershed for the remaining straw below. This straw which had not retted was covered by a dusty material which was probably composed largely of mold spores.

The forming of this water-proof layer over the horizontally placed bundles suggested that perhaps a more uniform retting could

be obtained by setting the tanks on end so the bundles of flax straw would be in a vertical position and which would allow the water to run down the stems from above. In the first trials several small bundles were used but it was found that retting was not uniform, the top or upper portions and the outside portion towards the opening where the water was applied retted while the other portions were only partially retted. A plentiful supply of moisture in the form of a fine spray was applied but it did not follow all of the stems down to the root end but rather developed small waterways on the side towards the opening previously mentioned. In spraying the water on the bundles most of the water was applied to the top, directing the water straight downward. Also, the outside of the bundles towards the opening was given a supply of moisture while the other side or back side of the bundles remained more or less dry.

As a final resort it was decided to place one large bundle of straw in each tank and select samples of straw from the inside portion to work up. The large bundles were treated as previously described and at the end of two weeks were opened up so as to select samples. It was found that the straw was not retted uniformly, some stems being only slightly retted throughout most of their length while others were fairly well retted. Also, a larger portion of the stems were retted above the middle as was previously found to be the case.

The unsuccessful results obtained by dew retting might be attributed to an insufficient supply of moisture to support a

luxuriant growth of organisms at all times. Because the straw was not packed tightly in a large container, it did not have the moisture retaining power and also probably did not have as good an opportunity to absorb as much moisture because of the greater ease with which the water percolated down through between the stems.

Ruschmann (71) states that under favorable conditions a first-class product can be obtained which would tend to verify the above assumptions as regards the causes to explain the unsatisfactory results obtained.

The Ochmann process has given rather satisfactory results in Europe and according to Carbone (20) in 1930 some of the retting establishments were using the pumps formerly used in the Coussine process to carry out the aeration of the water shower in this process.

SUMMARY

Various chemical solutions were used at different concentrations and temperatures, for varying lengths of time in an attempt to find one or more that would effectively ret flax. Acids, bases and inorganic salts were not satisfactory. Sodium and ammonium oxalates yielded a bleached fiber of rather low quality.

Degumming with soaps gave the most satisfactory results. A creamy white to white fiber of fairly good quality was produced. The fiber, however, was not uniform in color and usually not white enough to eliminate bleaching entirely where bleached yarns were

desired. It was found difficult to remove the adhering soapy materials from the degummed fiber. Washing with warm water gave better results than with cold water, dilute acid, alkali or ammonium oxalate solutions. The amount of water necessary to remove most of the adhering soapy materials was prohibitive from an economic standpoint.

The strength of soap retted fiber did not decrease by remaining in the degumming bath a longer time than was necessary to completely loosen the shives from the fiber. The strength tests were considerably lower than for fiber from natural water retted straw but were equal to water retted decorticated fiber. This supports the belief that decorticating unretted straw by present methods weakens the fiber.

Chemical retting processes reported in this paper were unsatisfactory and do not warrant their adoption in place of the natural warm water tank retting that is generally being used at the present time.

Flax straw retted by the warm water tank method gave a strong, bright colored fiber of high quality.

Attempts to ret flax in old retting liquor were unsuccessful. The retting process was prolonged and a very poor quality fiber was produced.

Biological degumming of decorticated fiber gave unsatisfactory results. The quality of the degummed fiber was poor. It was dull colored, dry and weak. No standard test can be used to

determine accurately when retting is completed thereby making it difficult to stop the process at the exact time.

Dew retting in a closed chamber gave unsatisfactory results. Retting was not uniform, gave a dark colored fiber and required at least a week longer than natural warm water retting in addition to requiring more labor and expense.

1. Anderson, D. B. The Structure and formation of bast fibers in flax. *Science*, 64, No. 1646:64-65, 1926.
2. Anonymous. Blachon and Peretmere Process. *Irish Textile Journal*, 22:111, 1907.
3. ——— The Rogers Process (not exact title). *Irish Textile Journal*, 22:139, Oct. 15, 1907.
4. ——— The Rosseau Process. *Irish Textile Journal*, 23:83, 1908.
5. ——— The Cheveline Process. *Textile World Record*, 44:85, Oct. 1912.
6. ——— Chemical retting with milk casein (not exact title). *Textile World Record*, 49:127, April, 1915.
7. Baird, J. C. and W. Duff. Investigations during 1928 and 1929 into the effects of flax water on fish life. *Jour. Ministry Agr. Northern Ireland*, 3:49-61, 1931.
8. Barker, M. F. and J. V. Eyre. A chemical study of the flax plant. II.--The variation of the constituents of flax seed with maturity. *Linen Indus. Res. Assoc. Memoir*, 15:11, 1923.
9. Bazzocchi, Antonio. Sulla macerazione industriale delle piante tessili. *Stucchi e ceretti*, Milano, 1921.
10. Behrens, J. Ueber die Taurotte von Flachs und Hanf. *Cent. f. Bakt. II. Abt.* 10:524-530, 1903.
11. Body, C. A. Million dollar linen manufacturing plant. *Chicago Tribune*, part 1, p. 20, Sept. 24, 1933.
12. Bradshaw, J. Method of retting flax straw. *Textile American*, P. 25, Sept. 1912.
13. Bredeman, A. M. *Bacillus amylobacter* in morphologischer und systematischer Beziehung. *Centralbl fur Bakt. II. Abt.* 23:385, 1909.
14. Bredeman, G. Einfluss Der Erntezeit auf die Beschaffenheit der leinsaat. *Faserforschung*, 4:234-243, 1925.
15. Carter, H. A. Microbiological retting of flax, hemp, and ramie. *Textile Recorder*, 37, No. 437,:127, etc. 1919.

16. Canadian Ministry of Agriculture Report. Flax retting in Canada, p. 65, 1919.
17. Carbone, D. La macerazione industriale delle piante tessili col "*Bacillus felsineus*". 2. Aufl., S. 159, Milano, 1926.
18. ——— Sulla macerazione delle piante tessili. Comunicazione al XIII Congresso Internazionale d'Agricoltura. Roma. Maggio-giugno, 1927.
19. ——— Lo stato attuale della macerazione microbiologica industriale delle piante tessili. Comunicazione fatta al Congresso Internazionale di Chimica Tessile e Coloristica. Milano, Maggio, 1930.
20. ——— La macerazione del lino a Courtrai. Giornale di agricoltura della Domenica, (5 Feb.) Bd. IX, 6s, mit 5 abb., 1931.
21. Davis, R. L. Flax-stem anatomy in relation to retting. U. S. D. A. Dept. Bul. No. 1185, 1923.
22. Demytt, Leo. Supt. State Flax Industry, Salem, Oregon. Letter dated Feb. 19, 1934.
23. Diakonov, N. J. Flax Retting. I. Effect of retting on yield of fiber. Jour. of the Textile Institute, 16:A218, 1925.
24. Dillman, A. C. Daily growth and oil content of flax seeds. Jour. Agr. Res., 37:357-377, 1928.
25. Dodge, C. R. A report on flax culture for seed and fiber in Europe and America. U. S. D. A. Report No. 10, 1898.
26. Durant, Albert. The flax scutching plant of Goderville. Can. Dept. of Agr. Pamphlet No. 1;4-11, 1915.
27. Ehrlich, F. Chemistry of pectin and its relation to the formation of incrustations of cellulose. I. (trans. title) Cellulosechemie, 11:140-151, 1930.
28. Eyre, J. V. and E. A. Fisher. Some considerations affecting the growth of linseed as a farm crop in England. Jour. Agr. Science, 7:134, 1915.
29. Eyre, J. V. and C. R. Nodder. An experimental study of flax retting. I. The development of acidity during the retting of flax: Its interpretation and technical significance. Jour. Textile Institute, 15, No. 5, May, 1924.
30. Flieg, O. Die Harnstoffroste. Faserforschung, 4:131-141, 1924.
31. Geiss, Reinhold. Suddeutsche Rohstoff. Verwertung, 6 Maffeistrasse, Munich, Bavaria, 1932. (unpatented).

32. Hacker, W. Retting baths for spinning fibers. (trans. title). *Faserforschung*, 5:153-154, 1926.
33. Hauman, M. L. Etude microbiologique et chimique du rouissage aerobique du lin. *Annales de L'Institut Pasteur*, 16:379-385, 1902.
34. Herzog, R. O. Technologie der Textilfasern. Der Flachs. Band 5 (1): Muller, W. Die Aufbereitung des Flachses. Julius Springer, Berlin, 1930.
35. Hoffmeister, C. Zur analyse des Flachswachses. *Chem. Ber.* 36:1047, 1903.
36. Huntley, F. A. Flax fiber investigations. Wash. State Agr. College Expt. Station Bul. No. 33, 1898.
37. Hutchinson, R. J. Report of the Chief Officer, Div. of Economic Fibre Production. *Can. Bulletin*, 1931.
38. Kayser, E. and H. Delaval. Contribution a l'etude du rouissage. *Bull. Soc. d'encour.*, 132:277-296, 1920.
39. Kind, W. Pectin contents of flax. (not exact trans. title). *Melliand's Textilber*, 7:872, 1926.
40. Korzheniovskii, G. A. Cooking flax yarn with sodium oxalate. (not exact trans. title). *Chimie and Industrie*, 24:946, 1930.
41. Kraus, Paul. Retting bast fibers. *Jour. Soc. Chem. Ind.* 38:169A, 1919.
42. ——— Retting bast fibers (trans title). *Z. Angew. Chem.* 32:160-162; 326-327, 1919.
43. ——— Retting bast fibers. V. (trans. title). *Z. Angew. Chem.*, 33:277-278, 1920.
44. ——— Isolation of bast fibers. IV. (trans title) *Z. Angew. Chem.*, 33:102-103, 1920.
45. Kranzlin, Gottfried. Rostwasser. *Faserforschung*, 2:126-150, 1922.
46. Lazarkevitch, N. A. Le Lin. sa culture et son industrie dans l'Europe occidentale. Gauthier-Villars Et. Co., Paris, 1925.
47. Lowry, G. A. A new era for flax. *Textile Recorder*, 47: 35-36, 1929.
48. Makrinov, I. A. Sur Un Nouveau microorganisme provoquant la fermentation de l'amidon et des materieres pectiques. *Arch. des Sci. Biol. Inst. Imp. Med. Exp. Petrograd*, 18:440-452, 1915.

49. _____ Anaerobic pectin fermentation. (trans. title)
Zentr. Bakt. Parasitenk. II. Abt. 85:339-348, 1932.
50. McColl, J. Copy of letter dated Feb. 2, 1918 to Mr. L. H. Dewey, U. S. D. A., Washington, D. C.
51. Muller, W. Rolling retted flax. (Trans. title) Leipziger Monatschrift, 39, S-N 1:16, 1924.
52. _____ Neue wege zur fasergewinnung. II. Jahrgang, Hett 7, Jan. 21, 1932.
53. Omeliansky, V., et M. Kononoff. Sur une methode de culture du bacille du rouissage du lin. Compt. Rend. Acad. Sci. Paris, 183:919-921, 1926.
54. Opitz, K. and A. V. Pander. Die Beeinflussung der saatgutbeschaffenheit des leins durch erntezeit und aussaatmenge. Faserforschung, 3:234-240, 1923.
55. Parisi, E. and Z. Tomassini. Pectic substances in the macerated stalks of flax. (trans. title--Chem. Abs.) Ann. Chim. Applicata, 21:559-563, 1931.
56. Pritchard, R. L. Copy of letter dated June 25, 1909 to Mr. L. H. Dewey, U. S. D. A., Washington, D. C.
57. Pritchard Flax Fibre & Pulp Co., Ltd., The. 120 St. Vincent Street, Glasgow, C 2. Flax and the Pritchard Process. 1929 (?).
58. Robinson, B. B. Report on observations made on different phases of the flax industry in Europe in the summer of 1930. December, 1930. Unpublished.
59. _____ The time to harvest fiber flax. U. S. D. A. Tech. Bul. No. 236, 1931.
60. _____ Copy of letter dated April 3, 1933 from Mr. Leo Demytt, State Flax Industry, Salem, Oregon.
61. _____ Report on sea kelp soap retting. 1933. Unpublished.
62. _____ and H. T. Johnston. Soap retting of fiber flax. Annual Report of Agronomic Work for 1933 by B. B. Robinson. Unpublished.
63. _____ and R. M. Snyder. Flax investigations at the Michigan Station. Mich. Station Report, p. 16, 1926.
64. Rossi, G. Industrial retting of textile plants by microbiological action. Int. Review of the Science and Practice of Agriculture, Year VII, No. 8:1067-1075, 1916.

65. Ruschmann, G. Degree and importance of the acid formation in biological retting. (trans. title). *Faserforschung*, 1:33-46, 1921.
66. _____ Retting Process--Ochmann's. (trans title and abs.) *Faserforschung*, 2:285-289, 1922.
67. _____ Taurosterreger. *Faserforschung*, 3:22-40, 1923.
68. _____ Grundlagen der Roste, Buch. *Faserforschung*, herausg. v. F. Tobler. Bd. 1, Leipzig, 1923.
69. _____ Entwertung des Schwungflachses durch Mikroorganismen. *Faserforschung*, 3:131-161, 1923.
70. _____ Fehlerhafte Roste. *Faserforschung*, 3:314-318, 1923.
71. _____ Our present knowledge of the retting process of flax. Translated by W. S. Davis. *Jour. Textile Institute*, 15, T61-74 and T104-114, 1924.
72. _____ Uber Rostfehler. *Leipz. Monatsschr. Textilind.* Sondernummer, S. 15, 1924.
73. Selligren, G. Inhemiska fibervaxter I. *Linum usitatissimum*, lin. *Kungl. Landbruks-Akademiens Handlingar och Tidskrift*, 1920.
74. Semmes, H. H. Memo enclosed in letter of Feb. 16, 1934 to Mr. J. W. Lewis, State Flax Industry, Salem, Oregon.
75. Smith, F. Annual Report of the Fiber Expert. Report of Dept. of Agr. Bengal, 38-60, 1929.
76. Snyder, Harry. Water for retting flax. *Minn. Agr. Exp. Sta. Bulletin No. 63*, 1899.
77. Stampa, G. Present condition of industrial microbiological retting of textile plants. *International Review of Agriculture*, 22, No. 2:63-74T, 1931.
78. Stormer, K. Ueber die Wasseroste des flachses. *Chem. Zent.* 76:41, 1905.
79. Tanner, F. Microbiology of flax retting. *Bot. Gaz.* 74: 174-185, 1922.
80. Tobler, F. The Carbone Ret in Germany. *Jour. of the Textile Institute*, 14, No. 4:A71, 1923.
81. _____ Bemerkungen und Versuche zur Harnstoffroste. *Faserforschung*, 4:141-145, 1924.
82. _____ Russian Flax Literature for 1925. (Chem. Abs.) *Faserforschung*, 5:261-268, 1926.

83. Trevethick, A., B. B. Robinson, and R. M. Snyder. Studies in flax retting. Mich. Tech. Bul. No. 95, 1928.
84. Troitzkii, B. V. The enzymes in aerobic and anaerobic fermentation of pectic substances. (trans. title--Chem. Abs.) Arkh. Biol. Nauk. 29:315-331, 1929.
85. Vansteenkiste, Constant. The rational preparation of flax fibre. Brochure. No. 205, March 1, 1929, Ypres, Belgium. Trans. by T. J. Crouch.
86. van Tieghem, . Sur la fermentation de la cellulose. Bul. Soc. Bot. de France, 26:25, 1879.
87. Watson, W. W. Copy of letter and circular to Ministry of Agriculture, Washington, D. C., March 29, 1932.
88. Winogradsky, S. Sur le rouissage du lin et son agent microbien. Comp. Rend. de l'Acad. des Sci. de France, 121:742-745, 1895. Translated by R. L. Davis.
89. Wyant, Zae Northrupt. Some bacteriological problems involved in the retting of flax. Abs. Bact., 5:208, 1921.
90. Zakochtchikov, A. P., I. A. Kohyeniou, and E. Ykioiuhkin. Experiments on the application of neutral salts on the cottonization of flax. (trans. title--Chem. Abstracts). Izvestiya Tekstilnoi Prom. i Torgovli, 9:91-95, 1930.