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A Photographic Study of the Effects of Beating on Fiber Structure

Gene D. Anderson
Western Michigan College

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A PHOTOGRAPHIC STUDY
OF THE
EFFECTS OF BEATING ON FIBER STRUCTURE

A

Dissertation Submitted to the Faculty

of

Western Michigan College

Kalamazoo, Michigan

by

Gene D. Anderson

In Partial Fulfillment of a Prerequisite for the Degree

of

Bachelor of Science

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Summary

Because beating is the most fundamental, but highly important, process in the paper industry, some of the changes in fiber structure that occur during beating have been investigated. A literature survey is also presented concerning a number of related investigations of fiber structure.

The experimental results indicate that fibrillation begins very shortly after the beating process is started. As beating progresses, the primary cell wall is removed, and the specific area of the fiber increases greatly when the watery medium encounters the secondary lamella area of the fiber. Continued beating shows that the action is a mechanical one, in that the fibers become bruised, brushed and cut into shorter lengths. The mechanical agitation of the Valley Beater unraveled the small fibrils that are wrapped spirally around the fiber. Prolonged beating eventually detached the fibrils and destroyed fiber structure entirely. After five hours beating, all that remained was a mass of fibrils that readily formed a mat on the slide made for microscopic study. These results compare favorably with published data.

The investigation did not prove that mucilage formed after extensive beating is amorphous. It appears as though the mucilage consists of fibrils and micro-fibrils, yet, equipment limitations prevented further work along these lines.

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LITERATURE SURVEY

A PHOTOGRAPHIC STUDY
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EFFECTS OF BEATING ON FIBER STRUCTURE

Introduction

The pulp and paper industry has long recognized the need, in fact the necessity, of pulp evaluation techniques by which predictions can be made regarding the expected behavior of pulps during processing, and also of the subsequent expected properties of the products in which the pulps are to be used. These predictions may be used by the pulp producer as a basis for controlling and altering the pulping process, and by the papermaker as a quick and dependable guide in applying the various process treatments to the many pulps available for paper manufacture.

Many research and development studies concerned with pulps have resulted in considerable publication of literature, which indicates there is considerable interest in the search for acceptable predictive techniques. Yet, despite this interest and activity, progress has not proceeded to the extent that dependable predictions can be established in all cases. It has been found that partial or even complete failure often resulted from predictions based on present techniques. For instance, many a papermaker has had the experience of producing low-strength paper from a pulp containing equally as high strength tests as a previously used pulp from which strong paper was made. The papermaker then felt the pulp was at fault, yet a check with the pulp manufacturer revealed the pulping process had not been changed. The question then naturally arises, is it the test, is it the pulp, or is it the papermaker who is at fault? It follows then, that methods to determine and improve prediction of the expected behavior of pulps as they are transformed into useful products is still a pressing problem to the paper industry.

With such a problem, which is always present, regardless of the process or the nature of the end use of the paper, it was felt that a study of fiber structure and its changes as beating progresses would offer fundamental, but very important, knowledge that should be at the fingertips of any paper technologist.

Basic Raw Material

Cellulose is the basic raw material for papermaking and occurs in the form of fibers in a varieyt of growing plants. Cellulose is a carbohydrate which comprises the major portion of the wall tissue of several plant cells. The cellulose of the most interest to the papermaker is found as wood fibers, where it is not found pure but is associated with lignin, hemicelluloses, and other substances. The approximate quantity of cellulose found in spruce, perhaps the most widely used tree for papermaking, is 60 per cent. ?

Cellulose is a white, fibrous substance insoluble in water and organic solvents. It contains a very high tensile strength which makes it especially desirable as a component of paper. The condensed formula of cellulose is $C_6H_{10}O_5$, which at once suggests its relation to glucose, the formula of which is $C_6H_{12}O_6$. Cellulose thus occurs as an anhydroglucose, $C_6H_{12}O_6 - H_2O \rightarrow C_6H_{10}O_5$.

Cellulose is degraded by acids to a product of lower molecular weight which is known as hydrocellulose. If the action of acids is carried far enough, however, the fibers are converted into a white or cream colored powder. If the process is carried out under extreme drastic conditions, the end products are simple sugars. It is not the general practice to allow the hydrolysis to proceed to this extent, however. When cellulose fibers are subjected to mild acid conditions they retain their fibrous structure, although the solubility in water and alkali is increased and the tensile strength of the fibers is reduced. Heat, however, will have a more degrading effect on cellulose than mild acids or oxidizing agents (1). Fibers which have been dried at excessively high temperatures tend to lose some of their papermaking qualities. It is impossible to replace these desirable properties by merely putting moisture back into the fibers. The effect of heat is primarily due to dehydration, which if carried far enough, results in an appreciable loss in hygroscopicity and swelling ability of the fibers (2). The loss in swelling ability resulting from prolonged heating of fibers at 100° C. may be as high as 50 per cent (3). In addition, fibers heated at temperatures in excess of 200° C. for long periods of time lose their original structure (4). It is quite evident, therefore, that when cellulose fibers are heated at elevated temperatures they become fragile and have considerably reduced tensile strength.

Morphology of Cellulose Fibers

Cellulose fibers used for making paper are slender, capsular-shaped bodies. The average length is one millimeter for hardwoods and about three millimeters for softwoods. In general, the length of the fibers is about 100 times their respective diameters. Some fibers taper to pointed ends, some have rounded ends, and others are exceedingly blunt. Some fibers are thick-walled with slender cavities and others are thin-walled with large cavities. Thin walled fibers are known as springwood and the thick-walled as summerwood. In chemical pulping, springwood fibers generally collapse into ribbon-like structures, whereas the thicker walled summerwood fibers usually remain inflated (5). It is assumed, therefore, that springwood fibers tend to make a sheet of paper that is more flexible than paper made from summerwood fibers. Another reason why springwood fibers produce a more flexible sheet is because they are more abundantly provided with 'pits', which makes them considerably weaker.

Construction of Fibers

According to Clark (6), the unit cell of a cellulose crystal, first postulated by Sponsler and Doree (7), and elaborated upon by Clark, is formed by four glucose groups polymerized together. To form the cellulose molecule 50 to

200 of these groups are bound end to end through oxygen linkages very much like an ordinary chain. These rodlike molecules are combined together like a bundle of twigs in groups of about 76, except there is some doubt about their ends being in line to form the unit cellulose crystallite. The crystallites in turn are bound together in numbers to form the fibrillae, which are arranged spirally in layers to form the fiber, each layer having its fibrillae parallel to those in each layer at an angle to those in the previous layer. The innermost layers have the fibrillae lying almost parallel to the axis of the fiber and the outermost layers have the fibrillae progressively lying more and more circumferentially like bands around the fiber. This type of construction was also reported by Spier (8) and Ludtke (9).

Weil (10), in an article published in 1935, put forth the following theoretical concept of the structure of a fiber.

I. The center of the fiber is occupied by the lumen, or a central cavity, which is surrounded by the tertiary lamella. It is probable that the lumen was filled with protoplasm during the early stages of the fiber growth and that the tertiary lamella was a wall which protected this very delicate substance against any injurious influences from the outside. The tertiary lamella is not cellulosic in nature, probably furfuroidal in character, and resembles a very fine skin.

II. Following is the secondary lamella, which is arranged in several layers and divided by concentric rings of skine substance. When one layer is removed, it exposes the layer under it, etc. It is not unlike hoses of different diameters, one inside the other. The different layers may represent different periods of growth and these growths are separated by skins similar to the skins of an ordinary onion. The periods of growth are not a matter of years, but of days, and may possibly represent the daily growth of the fiber.

III. The layers of the secondary lamella are longitudinally subdivided by fine skins into strips which are spirally wound around the axis of the fiber. The strips are arranged to the fiber with the greatest possible strength by being wound alternately right and left hand spirals. The angle of pitch of the strips is not known, but it seems that the innermost strips next to the lumen are almost parallel to the axis of the fiber, while the strips extending outward have a progressively smaller pitch. This is in accord with the work Clark (6) had previously done.

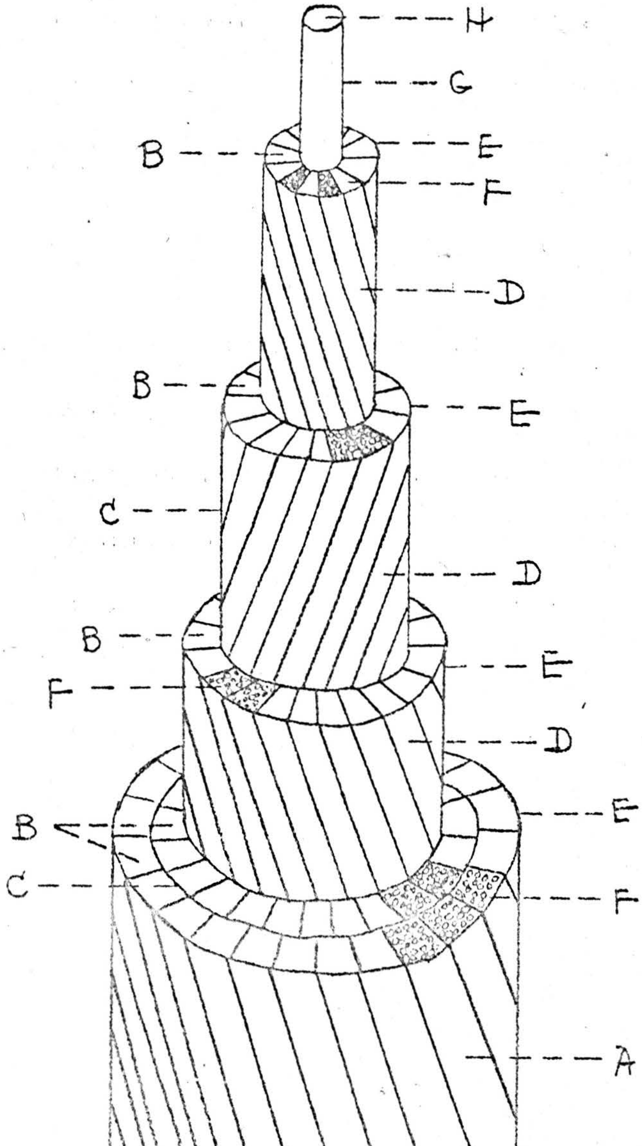
IV. The strips are built of fibrils, which are the smallest microscopically visible building units of the fiber. Probably the fibrils are separated by fine skins although some investigators believe they are cemented together by a colloidal inter-fibrous substance. The fibrils again are said to be transversely sub-divided by fine skins into still finer units known as fusiform bodies, or crystallites. The fibrils are cellulosic in nature.

V. The secondary lamella is covered by a primary lamella which is also of skin substance. The appearance of a bead-like necklace in a fiber when swollen in cuprammonium reagent may be proof of the existence of a more resistant membrane. Weil (10) also believes the fiber is composed of a strong skin skeleton where all skin elements are grown together. The skin skeleton is of a substance which is furfuroidal in character and spaces of the skeleton are filled with cellulose. The total amount of skin substance is very small, probably no more than 0.2-0.5% of the total fiber substance. The mere presence of the skin indicates the fiber is not homogeneous, which is an important factor in the beating of pulp.

VI. Included in the fiber also are fusiform bodies, which are of cellulose crystals, and the crystals of glucose groups. The unit cell is composed of a crystal which is formed by four glucose groups of size about 10X8X8 Angstrom units (A). The molecule, or micell, is composed of chains of from 30 to 50 glucose units which are bunched together in groups of from 40 to 60 chains. The size of the micell may be from 30-50 by 3-5 millimicron. About 75 of these micells form a crystallite of which the size is about 0.3X8X0.5 micron. Twenty to 100 of the fusiform bodies, when put one after the other and covered with skin, form a fibril, of which the dimensions are 0.3-0.8 by 15-16 micron, while the dimensions of the ultimate fiber, formed by 10-100 fibrils approximates 200-2000X20-30 micron. Figure 1, page 7-A, is a drawing of a structural model of a fiber cell.

The fact that the earlier work done on fibril dimensions was accomplished by means of the ordinary optical microscope gives rise to considerable question, since this type microscope has a limiting resolution in a range less than the dimensions of the fibrils. Some of the early investigators reported diameters of fibrils in the neighborhood of 0.1

STRUCTURAL MODEL OF A WOOD FIBER CELL



— LEGEND —

A- Primary Lamella
B- Four layers of the secondary lamella
C- Tangential Membrane
D- Radial Membranes
E- Double Cross Elements
F- Fibrils
G- Tertiary Lamella
H- Lumen

FIG. 1

as being 0.1 to 0.5 micron in diameter, but pointed out that they are, in turn, built of microfibrils, variable in size, but averaging about one-fifth to one-tenth the size of the fibrils. More recent work done with the electron microscope indicates the presence of very small fibrils having diameters of 50-100 A. and even less, depending on the source of cellulose (14)(15). These dimensions can also be subject to some criticizm since the size of 50 A. is approaching the limiting resolution of the electron microscope (16). Such dimensions approach the size of the hypothetical cellulose crystallite. Clark reported that microfibrils are in the range of 0.002 micron in diameter.

Micro-Dissection of Paper Pulp Fibers

Fiber structure has been studied by Seifrig and Hock (23) by means of an instrument known as the Zeiss-Peterfi micromanipulator. The technique involved the use of a mechanical manipulator by means of which delicate but rigid glass needles were very carefully controlled.

When a paper-pulp fiber is viewed in direct light it is seen to be marked by many surface lines which may represent surface folds, or actual strands or fibrils. This striated appearance can be brought out very vividly in a dark field. The fibers of both beaten and unbeaten pulp show the same characteristic of striated or fibrillar structure, though more pronounced in beaten pulp. If a single fiber taken from a beaten pulp is subjected to lateral pressure and flattened, the striae become separated, and stand out as individual strands.

When a fiber is penetrated by two micro-needles and the needles are moved perpendicularly apart, the fiber is split lengthwise and in such a way that the inner torn surface is perfectly smooth. This indicates that a papermaking fiber is not a homogeneous mass but consists of linear strands which readily separate when the fiber is torn. It was noted during the experiments that it became more and more obvious that these fibrils were not as fine as the very delicate strands which were frequently seen during dissection. Very careful micromanipulation revealed that the fibrils are themselves bundles of fine threads, so delicate as to defy individual handling and accurate determination of their size and number. These secondary fibrils approach the limit of microscopic visibility. The diameter of the primary fibrils is estimated at 1.4 micron. When a much beaten fiber is carefully dissected so as to spread out most of the secondary fibrils, the primary fibrils lose their identity and the entire flattened surface of the fiber appears as a sheet of innumerable fine strands. The secondary fibrils are estimated by Seifrig and Hock (23) to be of the order of 0.1 to 0.3 micron in diameter.

The Effects of Beating on Cellulose Fibers

Cellulose fibers must be subjected to mechanical treatment before they can be made into paper. The treatment may be applied in a number of different ways and methods, but usually the action includes rubbing, brushing or bruising of the fiber. This mechanical agitation of course, is varied considerably, according to the type of paper that is to be made.

The term 'beating' is used in the paper industry to describe the operation of mechanically treating the fibers. Throughout the years many investigators have made numerous experiments in efforts to determine what effects beating has on the papermaking fiber. All investigators discovered that beating is probably the most fundamentally important process in papermaking for manufacturing nearly all grades of paper, regardless of their end use. Even though the process is so important and apparently widely studied, the original Hollander beater developed in the eighteenth century is still very important today. In fact, the Hollander beater has undergone but very few improvements during more than 200 years since its inception.

Cross and Bevan (17) were early investigators of beating and in their textbook published in 1920 stated that beating accomplishes three main things: the separation of the fibers into separate units, development of flexibility and softening of the fiber, and in most cases the fibers are reduced to an extent which varies according to the type paper to be made. These three things always occur during beating, however, other physical changes also take place and they are determined by the action of the knives of the beater roll. Such action results in the bruising and flattening of the ultimate fibers and in many cases they are further reduced by splitting longitudinally into smaller units called fibrillae. In some cases, as in the manufacture of high class rag paper, cigarette paper, bank notes, glassine, etc., many of the individual fibers are beaten beyond recognition.

For very strong papers, cutting is necessarily avoided as far as possible, the needed disintegrating being brought about by bruising or breaking, giving the ends of the fibers a broken, splayed, or tangled appearance, which adds to their felting or strength qualities.

Cotton fibers are split by the bruising action of the rollat the point of rupture, into fibrillae, producing an interlaced network or trellised appearance. Linen, on the other hand, is split into a bundle of longitudinal fibrillae forming a paint-brush-like end. This deformation of the fibers increases their strength giving qualities. If however, the fibers are cut clean at the edge, they produce a paper of inferior strength, due to their inability to interlace and grasp one another.

A change takes place in the structure of the fiber as a result of its contact with a watery medium; the continual beating and agitation causes the cell wall of the fiber to absorb water and pass into the condition of a gelatinous hydrate. The stuff is said to feel greasy, an effect that may be regarded as due to a solution of water by and in cellulose. In effect, Cross and Bevan (17) believed that a cellulose hydrate was formed during beating.

Older Theories of Beating

Up until the year of 1925 the theory of beating was known as the chemical theory. It was generally thought that during beating part of the cellulose fibers gradually passed into the condition of a gelatinous hydrate (19) (20) in which the cellulose in part combined chemically with water. The process is not unlike soda ash as it combines chemically with 10 parts of water to form the hydrate commonly known as washing soda. The explanation of beating was that the fibers were cut and fibrillated and also a cellulose hydrate or slime was formed around the fibers and particles, which when dried, cemented the fibers in a sheet of paper together. This was a very comfortable theory to the papermaker at that time, but it was proved that it had its shortcomings.

J. Bell (18) agreed with Clark (6) regarding the main parts of the structure of the fiber, but further stated that there is a twofold aim of the beating process:

1. To get the fibers in such a state of free suspension in water that they can be made into a well-felted sheet, strong enough in the wet condition to run at the necessary speed of the paper machine.
2. To impart uniformity of texture and finish, the desired bulk and air porosity, and the desired strength and stretch to the finished sheet. Bell also classified the beating operation results as follows:

a) Separation of the individual fibers from fiber bundles.
b) Cutting the fibers into shorter lengths.
c) Splitting the fibers lengthwise into fibrillae.
d) Removal of entrained air from the fiber.
e) Softening of the fiber.

f) Production of fiber debris and structureless slime of mucilage.

Strachan's Physical Theory

James Strachan (21) in 1926 successfully attacked the chemical theory. In his paper he proposed that the taking up of water by pulp should be termed imbibition and not hydration.

In his experiments he found that in drying both beaten and unbeaten pulp under carefully controlled conditions there was no evidence of any break in the curve relating moisture content to time and consequently no evidence of the formation of a hydrate in the strict chemical sense.

Strachan described the structure of the cellulose fiber very much as we know it today and then postulated that the layers of the fibrillae on the fiber are porous, allowing water to penetrate and cause the fiber to swell. The fiber, when subjected to beating action has the outer layer loosened and the surface becomes fibrillated. The fibers of the beaten stock are soft and adhere together on the wire more strongly than when unbeaten fibers are subjected to the same process, and under the action of the presses of the paper machine the fibrillated surfaces of the fibers are pressed into intimate contact, and the cohesion is finally completed on the calenders.

Campbell's Theory on Bonding

W. Boyd Campbell (22) feels that when fibers are immersed in water or placed in an atmosphere containing moisture, a layer of water is absorbed on every exposed crystal surface and a layer of hydrated cellulose is thus formed. When the OH groups combine with the water the attraction for the crystallites for each other is lowered and allows additional water to enter which produces further imbibition and swelling. Beating causes stressing and bending of the swollen and softened structure, and since the fiber is made more flexible, fibrillation occurs and a very much greater external surface is thus produced. The fibers adhere together when paper is made by the bonds caused by the large surface tension forces and the secondary valence forces. The degree of bonding depends on the flexibility of the fibers and the amount of surface exposed, both of which are dependent upon the degree of beating.

Properties of Suspensions of Beaten Fibers

It is unlikely that quite severe beating will cut the fiber much less than 0.2 millimeter in length. The specific surface of long fibrous bodies will be little affected by transverse cutting and much more by fibrillation. An average papermaking unbeaten fiber may have a specific surface of about 0.2 sq. m/gm. If the fiber is completely resolved into fibrillae this value might be increased to 7 sq. m/gm.

By calculation from the dimensions of the micellar unit it would seem to be possible for the specific surface to reach values of 1000 sq. m/gm. if complete resolution to micellar dimensions took place. It is highly improbable, however, that the external surface will be multiplied as much as 20 times by even a severe beating treatment. Fiber length changes can be measured but there is no practical method for determining the degree of fibrillation.

Bell (18) carried on his work on the gel properties of beaten fibers long enough to state that some fibers will be acceptable for papermaking while others do not lend themselves at all to the process. He found that wool does not fibrillate nor does it form a satisfactory sheet of paper. Silk and asbestos split up into fibrillae, felt well enough, and can be formed into sheets, however the paper is weak due to relatively free slippage of the fibers past one another. Vegetable fibers alone have the colloidal surface condition essential to the formation of a strong sheet. This condition is manifested in water but not in other non-polar solvents. It seems apparent, therefore, that the surface of beaten fibers must be composed of an altered film rendered adhesive in some way by the agency of the water.

Hydration

Soaking of the fiber when it is placed in water is accompanied by swelling whereby the surface of the fiber is increased as is also the ability to hold more water. When the fiber is given further treatment in the beater it undergoes certain changes which have been designated by Weil (10) as "hydration".

In beating, the outer layer or primary lamella is damaged or bruised by a gentle action. By this action the fibril bundles as well as the individual fibrils of the first layer are loosened. Continuation of this process will loosen up all the fibrillae and actually detach some of them.

It is possible to make a strong paper by fibrillation of the outer layers even when the inner layers are intact. The loosened fibrils, with their flexibility allow much better interweaving and produce a stronger sheet. The greater strength of a piece of paper made from carefully fibrillated fibers is due to the fact that more points of contact are created to develop friction and to decrease slippage of the fibers. The loosening up of a certain amount of the fibrillae or the fibrillation of the fibers to a certain depth, therefore, determines the paper to be made. To attain maximum strength the strong fiber core should be preserved and only part of the fiber fibrillated. Weil (10) also found that beating produces a structureless slime or mucilage. The production of this mucilage, which is colloid in the gel form, is very important. It is the development of this colloidal material which not only covers the surface of the fibers but to a certain extent is also absorbed by them. The pores and spaces between the fibrils and fibers are also filled so that when such material is felted into a sheet of paper and dried, the entire mat is cemented and slippage of the fibers is at a minimum. This gel develops only when cellulose is beaten in water, which tends to prove that the cellulose fiber at time of growing was a colloid in liquid form and formed into the fiber as we know it by coagulation.

Present Day Beliefs

Today, most chemists believe that no major chemical changes in the fiber occur during beating, since the x-ray diffraction pattern is not changed as the result of beating (16). The principal effects of beating appear to be physical. Some of the most important physical changes are: Fracture and partial removal of the primary cell wall of the fiber, decrease in fiber length, increase in fiber flexibility, formation of fibrils, (better known as fibrillation), and an increase in the external specific surface of the fiber.

Of fundamental importance is the fact that beating affects the interfiber bonding in a sheet of paper. Therefore, beating is one of the most important steps in the manufacture of paper, and it also determines the papermaking qualities of a given pulp.

The three principal theories of beating generally accepted today to explain the effects which are produced on pulp fibers during beating are:

1. The chemical theory, mentioned earlier and suggested by such early investigators as Cross and Bevan (17) and Schwalbe (19).

2. The physical theory as proposed by Strachan (4) in 1926.
3. The partial solubility theory proposed by Campbell (22) in 1932.

Other investigators have offered modifications and variations of the original theories.

The early chemical theory of beating has been abandoned today because it has been reasonably well proved that water does not combine with cellulose to form a true chemical hydrate. Therefore, the term "hydration" which is commonly used in describing beating effects, is a misnomer. Beating of pulp results in physical changes in the fibrous structure and is primarily a mechanical process. Some minor chemical changes may possibly occur as a result of the great increase in surface activity of the fiber and other changes of the fiber (24) (25).

Swelling of Pulp Fibers

Dry pulp fibers vary considerably in stiffness depending upon the type of fiber, however, all dry fibers have a tendency to be brittle. On the other hand, water soaked fibers are very flexible because they have absorbed water and are swollen. This makes the individual fibers soft and pliable, increases their elasticity, and decreases stiffness. The increase in fiber flexibility is very desirable because it prevents extended breakage of the fibers during beating. The increase in fiber pliability also increases the fibrillation of the pulp. This in turn increases the bonding area in the sheet, resulting in increased density and strength of resulting paper. Some investigators (26) report that certain fibers on beating may swell to twice their dry diameter.

Most fibers swell fairly rapidly when placed in a watery medium, although some difference is noted between fibers which have soaked in water for a long time and those which have soaked for only a few minutes. This difference in soaking time is important in some cases because it means that water has entered the fiber and changes it from a ribbon to a cylinder.

Pulp fibers take up water by both porous imbibition and capillary absorption. In the process of porous imbibition the water is taken up in preformed cavities such as pits, pores and the lumen of the fiber. In this process the mere filling of these areas with water does not alter the dimensions of the fiber. When water enters the small sub-microscopic pores of a fiber, however, the process of imbibition and swelling overlap. Absorption of water in this way loosens the fibrils and allows them to separate as the action of beating is carried out. As the fiber swells, more and more spaces are opened up and allows additional water to enter.

The ability of pulp fibers to swell is attributed to the hydroxyl groups present (27) since cellulose fibers will not swell in polar liquids unless the polar hydroxyl groups are present. Kress and Bialkowsky (24) measured the swelling of cellulose fibers in various liquids and found that only highly polar liquids induce swelling, whereas non-polar liquids resulted in very little, if any, swelling. They found that fibers in water resulted in 90% increase in volume but only an increase of 2% was noted when the fibers were placed in fuel oil.

Effect of Beating on The Primary Wall

On the outside of a natural fiber is a layer known as the primary wall. This wall is permeable to water but does not swell appreciably. In many cases the primary wall is partially removed from the pulp fibers before they reach the paper mill, inasmuch as cooking, bleaching, and washing tend to remove it during the pulping process (28). This primary wall resists the effects of beating (29) and in some cases extensive beating is required since there is enough of the primary wall left on the surface of some pulps to prevent the maximum adhesion of the fibers. Beating action, however, tends to break up and rub off whatever part of the primary wall which is left and exposes the layers of the secondary wall. It is the secondary wall that swells very greatly in the presence of water. When this occurs the secondary wall is no longer constricted and can swell to two or three times its original diameter (30).

Fibrillation

A very important effect of beating is the fraying out or fibrillation of the fibers. Fibrillation involves a loosening of the coarse fibrils and a raising of fine fibrillae on the surface of the fibrils (28). The fibrillae are for the most part visible under the electron microscope although they probably range in size down to units which are invisible. Strachan originally visualized the beating process as a development of a fine pile of fibrillae on the surface of the fibers quite similar to the pile on velvet. The presence of this fine fuzz-like material accounts for the very large surface of beaten fibers.

Most paper chemists now recognize the importance of fibrillation, although there has been considerable disagreement among many investigators regarding the dimensions of the fibrils produced during beating. Strachan (13) observed the presence of fibrils when examining the surface of relatively unbeaten fibers with an ordinary optical microscope and assumed they were bundles of microfibrils about 0.2 micron in diameter. Electron micrographs taken at a magnification of 6200X show the presence of fibrils 0.05 micron in width and even smaller (32). Clark (33) believes that beaten fibers are covered with a very fine fuzz or micellae from 50 to 100 Angstroms in width, and these in turn are probably covered with an even finer fuzz of molecular dimensions. It is thought that the finer fuzz is cemented to the body of the fiber on drying and does not reappear until the pulp is again beaten. Sears, (34) utilizing the electron microscope, found very fine fibrils which he estimated to be 500 Angstroms or less in width.

Because of fibrillation, it is known that beating will cause the bursting strength of paper to rise, but if beating is carried beyond a certain point, the fibers become shorter and more mashed, and no further strength can be developed. It is apparent then, that at some time during the beating cycle the effect of fibrillation is neutralized by the production of more and more short fiber and debris, at which point the bursting strength begins to decrease. If beating is carried far enough, the fiber structure is completely destroyed and an amorphous material is all that can be seen under the microscope.

EXPERIMENTAL OUTLINE

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Purpose

The primary purpose of experimental work connected with this experiment was to provide an acceptable means whereby fiber structural changes could be observed as beating time increased.

It was known that a study of this nature could be made with the ordinary laboratory microscope where a magnification up to 430 times could be obtained. Using the microscope for general viewing however, is somewhat tedious, and it was felt that photomicrographs would offer better material from which to draw any conclusions. Furthermore, photomicrographs present a more permanent record that can be kept in a notebook for a quick, handy reference.

Materials

The materials used for the investigation included the following: A softwood pulp, fairly easily beaten, a laboratory Valley beater, microscopic slides, a Bausch and Lomb Laboratory microscope, a Bausch and Lomb vertical camera adapted for use with the above named microscope, a dye to produce slides with good contrast, fine grain film, an enlarger for print making, special printing paper for this type photography, and chemicals for developing and printing the photographs.

Preliminary Investigation

Initial experiments were made on fully bleached Puget Sulphite, but results were not too encouraging. With this pulp, even after four hours beating the fibrillation was not complete. Though most of the fibers were cut into shorter lengths the structure was still visible. It was then decided that perhaps an easier beating pulp would be better for such a study. Therefore, a Finnish import glassine quality pulp, known as 'Tornator', was tried and much better results were obtained. The pulp was manufactured by utilizing a slow acid cooking process.

Again, some trouble was encountered when the various slides were stained, and it was found after a series of trials, that the stain providing the best slides was the stain Carbol-fuchsin, an alcoholic solution of fuchsin in ~~dehydrated alcohol~~ phenol. This stain colored the fibers a bright, transparent red that appeared to offer excellent specimens for close examination under the microscope, and subsequent photographing of the slides.

EXPERIMENTAL INVESTIGATION

EXPERIMENTAL INVESTIGATION

Beating and Preparation of Slides

Initial laboratory work consisted of subjecting the Tornator Unbleached Sulphite pulp to prolonged beating in the laboratory Valley beater. TAPPI Method T 200 m-45 was used during the beating, with slight modifications. A disintegrator was not used prior to beating since it was found that the pulp could be sufficiently broken up by agitation and circulation for five minutes in the beater with no weight on the bedplate lever arm. The sample withdrawn at the end of the five minute circulation period represented zero beating time. Similarly, samples were withdrawn, after addition of a 5500-gram weight to the bedplate lever are, at the following time intervals: Five, ten, 15, 20, 25, 30, 45, 60, 90, 105, 120, 150, 180, 240 and 300 minutes beating time. These various samples were then diluted to a consistency of 0.05% and microscopic slides were prepared. The Carbofuchsin stain, previously referred to, is quite fugitive to light so slides were kept in an airtight container until they were actually examined. It was thought that a more accurate examination could be made if all slides were stained at the same time, and microscopic observation could then follow, one plate after the other.

Photomicrography

To make photographs suitable for a study of the structural changes, a Bausch and Lomb vertical camera with a fixed plate distance was used to photograph the various slides. The camera consisted of a metal box with a fixed plate distance of ten inches. It was equipped with a front board which held part of a light-tight fitting for the microscope. Also fitted to the side of the camera, just below the shutter, was a side tube with an ocular. The side tube contained a slightly silvered prism whose purpose was to direct some of the light rays to the ocular where the image of the fibers could be examined exactly as it was focused on the plate of the camera. When the side tube ocular was correctly adjusted, focusing was much easier and more exact than if focusing was done with the projection made on ground glass. The film size for the camera was 3¼ by 4¼ inches.

Since the extension of the camera was but ten inches, the intensity of the light which fell on the plate made fast exposure possible. The shutter allowed numerous instantaneous exposure settings in addition to time and bulb. The instantaneous setting ranged from 1/200 to 1/10 of a second. By trial and error, it was found that the best negatives were obtained with an exposure of 1/25 second.

The magnification obtained with this camera was somewhat limited, but, in general, the negatives were fairly sharp, probably due to the short plate distance. It was felt that better evaluation of the fibers could be made if the finished pictures were enlarged, so all pictures were enlarged so that the final magnification was approximately 200 diameters. A number of photomicrographs were made in the course of the experiment and some of them can be seen in the following section.

Experimental Evidence and Conclusions

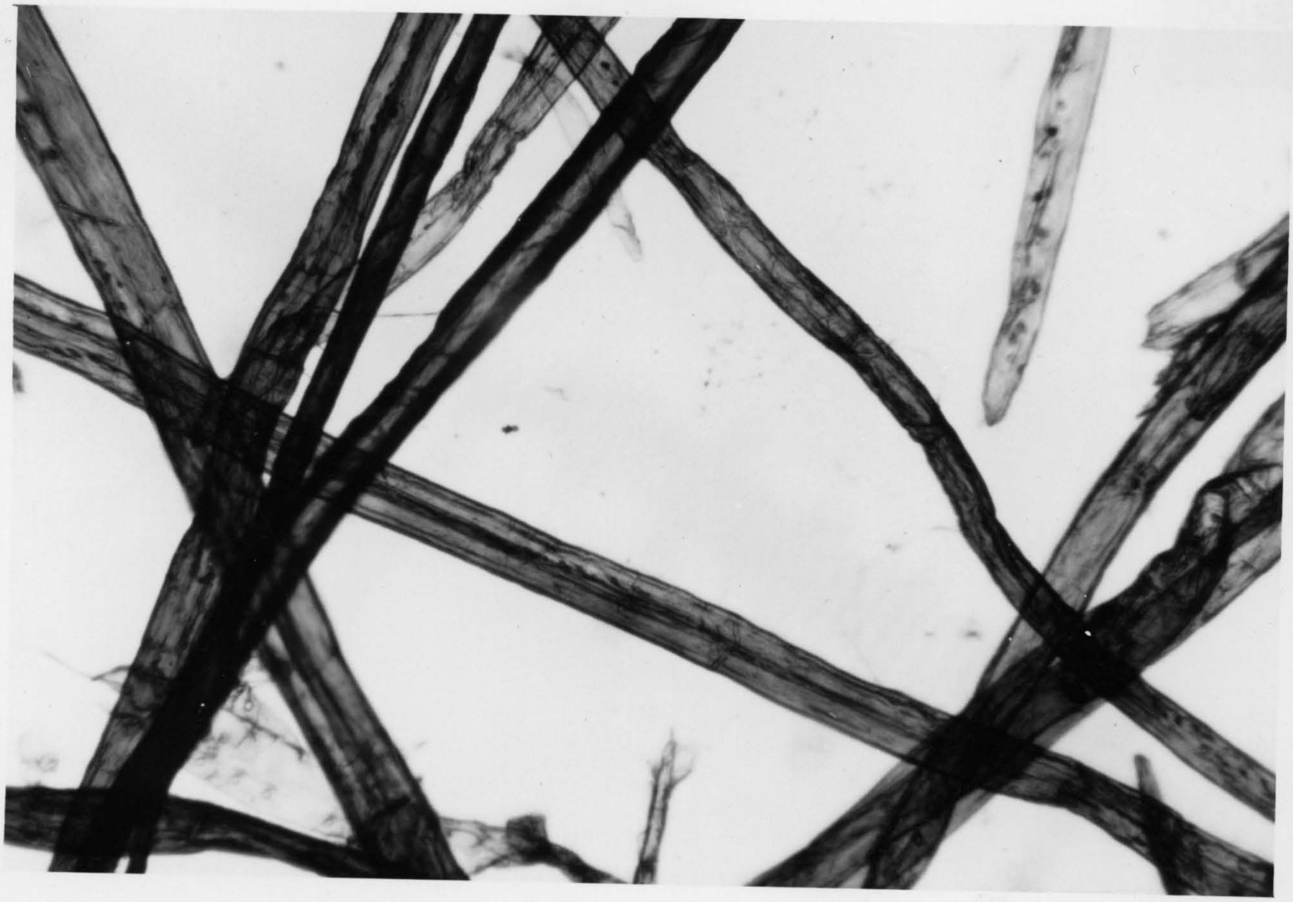
Examination of the photomicrographs made at zero beating time showed the individual fibers were separated from one another. The lumen was visible on some of them, as were a few bordered pits. The primary cell wall was smooth and spiral windings could be seen when the picture was enlarged better than 600 times. These lines were not entirely visible on all fibers, but their presence does indicate that they are probably fibril bundles, as reported by previous investigators (6)(10). It looked as though the angle the bundles followed around the fiber was perhaps 60 degrees from the fiber axis. Figure 2 shows the various properties just described. The photo was taken from fibers that had not undergone any beating action.

The slide prepared from pulp beaten for five minutes showed some of the fibers were cut into shorter lengths. In one instance, a tearing of the primary cell wall was noted. Also, it appeared as though the bordered pits may be weaknesses in the cell wall since tearing first occurred at these points. Even after this short beating time some fibrillation was in evidence.

The slides made at succeeding five or ten minute intervals were examined and about the only significant change noticed was additional cutting and continued fibrillation. As can be seen from the photograph made after one hour beating, (Figure 3) portions of the primary cell wall have become torn and separated from the secondary wall. The walls of the fibers are not smooth, but are 'fuzzy'. This small hair-like fuzz are evidently fibrils that have loosened from the secondary cell wall. It was especially noticeable that the fibrils are extremely small when compared to the width of the whole fiber. There was not enough magnification available to study very carefully the fibrils themselves, but when it is realized that the fibrils are just visible after a picture has been magnified more than 200 times, and idea of the actual size of the fibrils can be obtained. There is still considerable fiber structure remaining, however, the picture shows that most of the fibers have been cut into shorter lengths.

Further beating shows that at the end of two hours, considerable fibrillation had taken place and entanglement of the fibers can be seen (Figure 4).

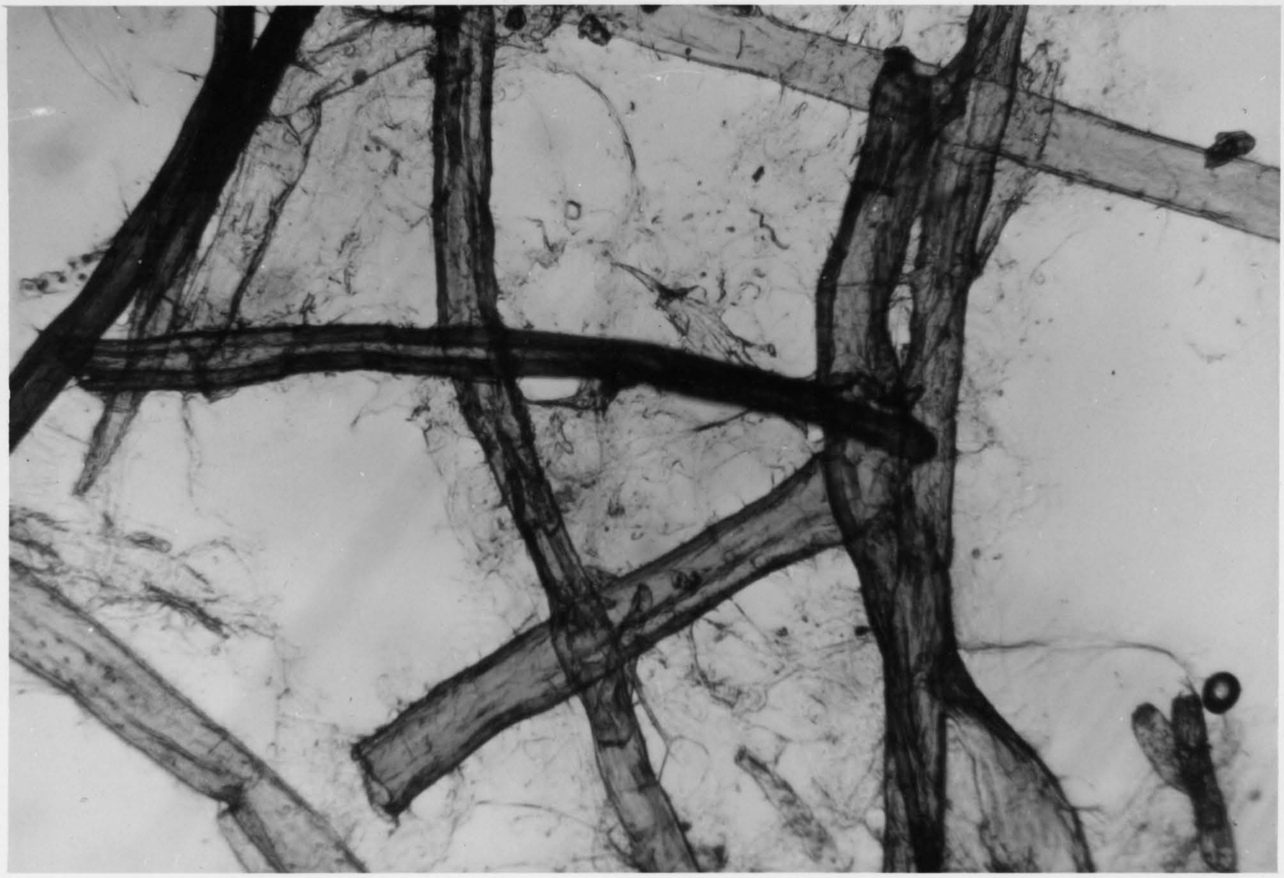
Figure 5 is a very interesting photograph. The primary cell wall of the fiber has been shattered by the beating action. Some of the fibrils can be seen to extend approximately 90 degrees from the axis of the fiber, but most of them appear to still be in bundles not yet unraveled.. Although the structure is not symmetrical, it can perhaps be compared to a short piece of stranded rope. If a section of such a rope is cut into a three inch length, held on one end and then thoroughly shaken, the individual strands tend to separate themselves from the adjacent ones and the rope becomes sort of 'bushy'. The picture (Fig. 5) appears to be taken just after the fibrils started to



200X

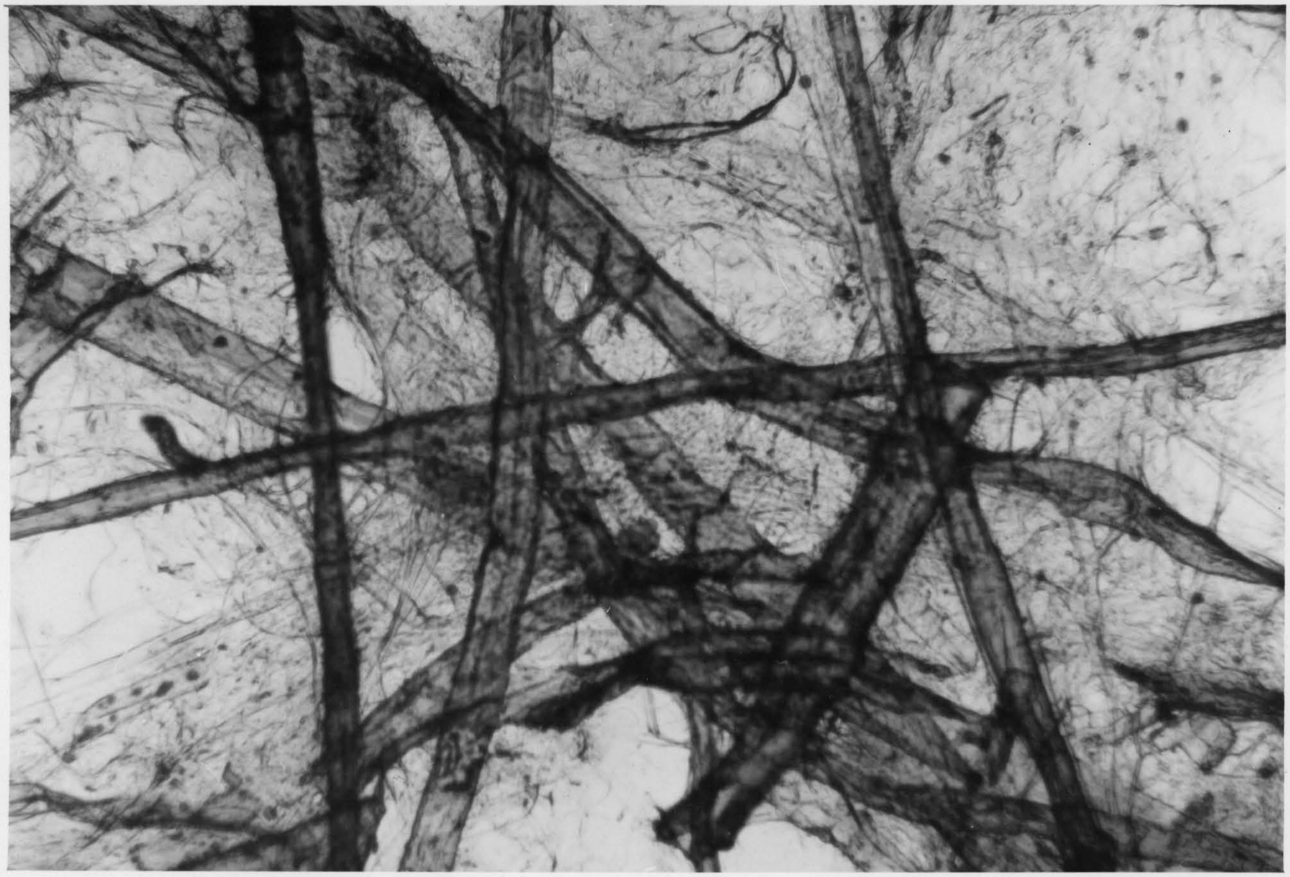
Figure 2.

Photomicrograph taken at zero beating time showing complete absence of fibrillation.



200X
Figure 3.

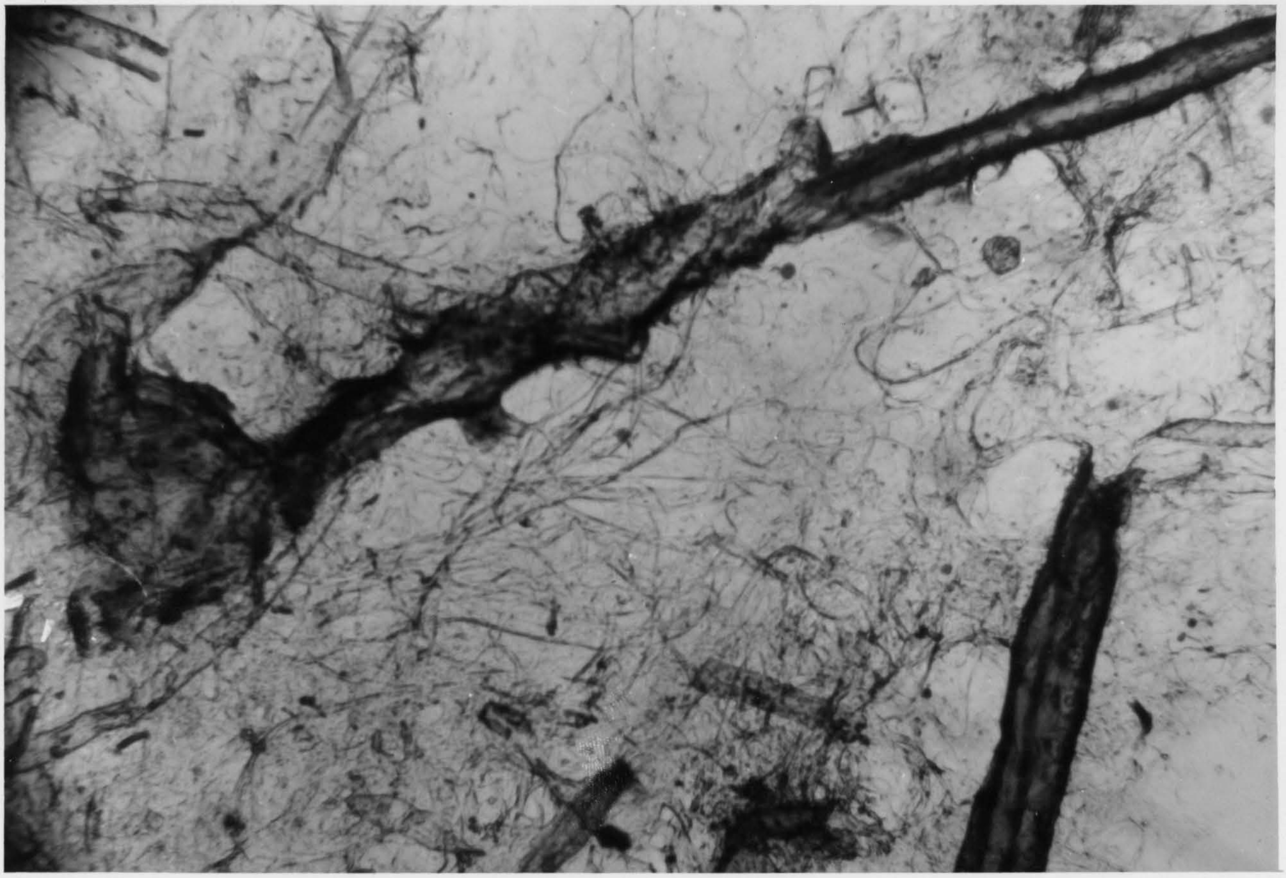
Photo taken after one hour beating time showing some cutting and fibrillation.



200X

Figure 4.

Photograph taken after two hours beating time showing continued cutting and fibrillation.



200X

Figure 5.

Photograph taken after four hours beating time. The angle at which the fibrils wrap around the fiber can be seen where fiber was caught between bedplate and flybar.

unravel and upon continued agitation and beating they probably became detached entirely from the fiber.

The photographs starting with Figure 3 show that the development of the degree of beating results from an increase in damaged fibers, and also an increase in the quantity of slime or mucilage formed. In the beater, this mucilage actually feels gel-like and slippery, with apparently no visible structure. One conclusion that seems evident, yet perhaps questionable, is that the slime is evidently composed of an entanglement of the finer fibrils and fibril bundles. If this is true, as is indicated from the experiment, the mucilage is not a structureless, colloidal mass, as was noted in several references read during the literature survey (10)(17)(18)(36).

Figure 6 shows fiber structure after six hour beating time. No fiber structure remains. All fibers have been reduced to very fine fibrils that formed an entangled mat when the microscopic slide was made. The pulp from which these fibers was taken did not have any body in the beater. When a handful was taken from the beater it was impossible to form a ball of pulp, a procedure which is often done in the beater room during commercial manufacture of paper.

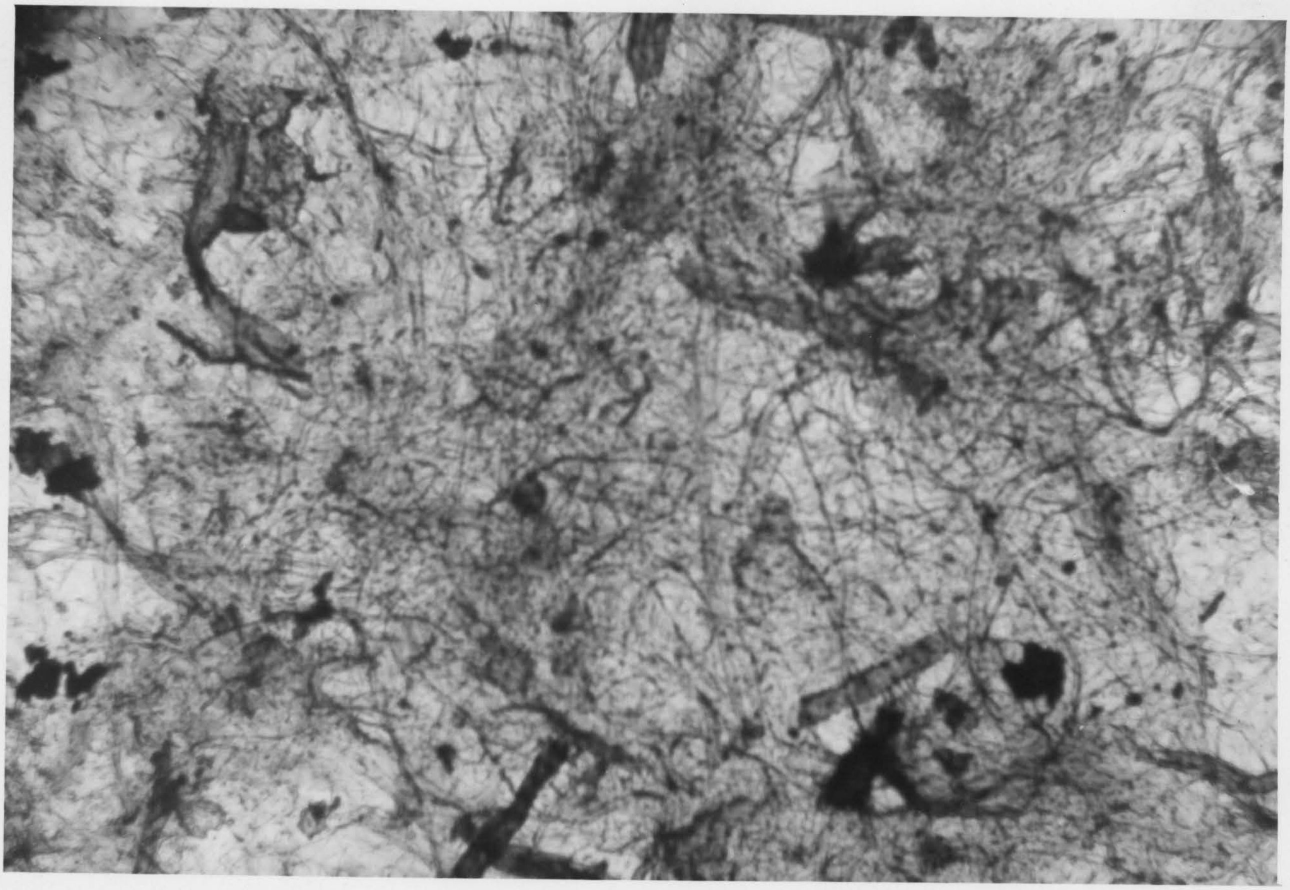
Some of the morphological details brought out in previous paragraphs can usually be recognized only on carefully beaten fibers. It appears that the fibers caught between the fly bars of the beater roll and the bed plate were squeezed and defibrillated immediately. This action, of course, varies in severity depending on how the fiber is caught in the tackle.

The fibrillation noted on the five minute beating time photograph evidently resulted from such action. On the other hand, some of the fibers remained unattacked even during considerably more beating. If beating is carried out long enough, however, all fibers are eventually attacked and eventually lose all semblance of structure. Figure 6 shows what happens to fiber structure upon prolonged beating.

Matters for Further Consideration

The results obtained as a result of this investigation agree very closely with the work done by many previous investigators. Yet, the many claims that the mucilage formed during beating is structureless, has not been fully substantiated by this work. Another avenue of approach for a study of structural changes of the fiber during the beating process might include a more lengthy study of this so called amorphous slime. Such a study might produce results that agree with previous work, or perhaps new light might be shed regarding this problem.

Gene D. Anderson



200X

Figure 6.

Photograph taken after five hours beating time showing complete absence of original structure.

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