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Microscopy of Newly Developed Paper Making Fibers

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By

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ABSTRACT

The more important of the newly developed fiberous paper making materials are bleached groundwood, West coast kraft, hardwood kraft, semichemical pulp and cotton linters. The weight factors of these fiberous materials have been determined by several different investigators, but their identification in a paper furnish is very difficult. The weight factors of cotton linters varies with the freeness of the sample, which can be determined by dying the fibers with a mixture of a direct orange and a direct blue dye. Differentiation can be made between bleached and unbleached groundwood with the Lofton-Merritt stain. Staining methods cannot be used to differentiate between soda, hardwood kraft, and neutral sulphite semi-chemical pulps. Kraft cooked semi-chemical pulp gives a deeper lignin reaction with stains. Cotton linters have a thicker cell wall than long staple cotton , and unbeaten cotton linters stain a deeper blue when stained with a mixture of a direct blue and a direct orange dye. Unless the furnish has been highly beaten, West coast kraft made from Douglas fir and western hemlock can be identified by the microscopical structure of their fibers.

With the perfection of new pulping and bleaching methods several new pulps are now being used in paper furnishes. Species of wood, which were used only to a limited extent in the past are now being used in many kinds of paper. (9) Among the more common of the new paper making fibers are bleached groundwood pulp, kraft pulp produced from western hemlock and douglas fir on the West coast, kraft pulp made from hardwoods, neutral sulfite semichemical pulp, kraft semichemical pulp, and cotton linters. Each of these new paper making fibers offers a problem of its own to the microscopist who finds any one of them in a sheet of paper of unknown furnish.

Groundwood pulp in a paper furnish is easily identified, but to determine if the groundwood is bleached, semi-bleached, or unbleached and the percentage of each that is in the sample is a task that cannot be accomplished through the use of standard, well-known microscopical methods.

Kraft fibers produced on the west coast from douglas fir or western hemlock can be identified by the structure of the fibers, but if the pulp is made from a western species of pine or is a highly beaten pulp, identification becomes difficult and uncertain. At one time almost all of the deciduous fibers which were found in a paper sample could be identified

as having been produced by a soda cook, but pulping of hardwoods by the kraft process, the semi-chemical process (both kraft and neutral sulfite cooks), and the sulfite process has made this identification impossible and has further complicated the work of the microscopist.

The microscopist may use several different procedures to aid him in the identification of the various pulping fibers which he may find in a paper furnish. The most common and the most widely used method of establishing the fiber furnish of a paper is to first pulp it. Second, the fibers are mounted on a microscope slide and differentially stained. Third, the fibers are counted, and the number of fibers of each kind of pulp found in the sample are multiplied by their appropriate weight factor, which transforms the fiber count into fiber weights. The fibers in the sample are then reported as the percentage of the total number of fibers in the sample by weight. (5) The weight factors which are used by most of the microscopists are listed in Table 1. This table also includes weight factors for some of the newer of the fibers used in paper making.

WEIGHT FACTORS

The weight factor of a pulp fiber is dependant on its

on its length and width, that is, the weight factor is proportional to the average weight per unit length of the fiber. There are two methods by which the weight factor of a fiber and its corrosponding grex, grams per one thousand meters, may be determined. The first and slower of the two is a microscopical method, and the second is a projection method, which takes about half as much time as the first method.

In the first or microscopical method a suspension of the fibers, containing about 0.038 grams of the fibers per liter of the suspension, is prepared. This suspension is well mixed and used to fill four large test tubes. The mixture in each test tube is used to prepare two slides. (4) In the preparation of the slides one drop of the suspension in a test tube is placed on each end of one of the slides; two slides being prepared from each test tube of the mixture--eight slides being prepared in all. The slides are dried on a hot plate at a temperature below eighty-five degrees centigrade. As soon as the fibers are completely dry, they are removed from the hot plate, and two drops of Herzberg stain are placed on each of the groups of fibers. The stain is allowed to remain in contact with the fibers for two minutes, at which time the excess stain is drained from the slide, care being taken to insure that no fibers are lost in the process. Next, the slides are placed under a dissecting microscope,

and the fibers are straightened out, being drawn toward the center of the slide and being arranged parellel to the slides long axis. The length and width of the fibers is measured, a total of two hundred fibers being used to determine the weight factor. If the eight slides do not contain two hundren fibers, more slides will have to be made up until a total of two hundred fibers can be counted. From these measureaments the average length and width of the fibers is determined from which the corresponding weight factor and grex is determined. This is a long and extremely tedious taking at least one hour or one and one half hours to complete. (18) It can only be applied accurately to fibers which have a well defined and simple shape.

In the optical projection method of determining the average fiber length and width with the corresponding grex, a water suspension of the fibers is made up, and a slide of the fibers is prepared in the same manner in which the slides are prepared for the microscopical determination of fiber lengths and widths. Instead of using a microscope, a microprojector is used as the magnifying instrument. A suitable microprojector would consist of a series of lenses which would project an image of the fibers which were placed on the stage to a screen. The projector should be able to magnify the

fibers at least fifty times. The screen should be divided into a series of concentric circles; the radius of each successive circle being no more than one half millimeter larger than the preceeding circle. The center circle should be divided into smaller divisions by means of a series of dots. (7) Before a measurement can be made on any pulp sample it is necessary to check the magnification and focus by project- the image of a standard of known length on the screen. Once the microprojector is focused and adjusted, the fibers are counted in the same as with a microscope.

In calculating the weight factor, the fiber is compared with a standard rag fiber, which is defined as having a weight factor of one and a grex of one and eight tenths grams per one thousand meters . (5)

If in a sample of paper whose fiber furnish is to be determined, there are fibers of unknown and their weight factors are not know, a fairly accurate weight factor can be determined by using an empirical formula, which gives a weight factor that can be used to determine the weight percentage of the unknown fibers which are in the sample. The tenative weight factor is determined by dividing the average width of the fibers in microns by the factor of twenty for rag fibers, thirty for coniferous fibers, and thirty-five for deciduous

or grass fibers. The weight factors determined in this manner are fairly consistant with the established factors, which are being used in fiber analysis.

There is some controversy on the subject of the effect that beating has on weight factors. Mr. Graff (9) used several different kinds of pulps, each beaten to three different freenesses, and combined them in all possible combinations. His conclusions were that the weight factor was not affected to any large degree by beating. Mr. Clark (5) does not agree completely with Mr. Graff on this point. He believes that the only connection between freeness and weight factors is very minor with the exception of the weight factors of cotton and of some of the bast fibers where the decrease in freeness is caused by the lengthwise splitting of the fibers. With other fibers which have little tendency to split lengthwise with beating, the decrease in freeness with beating time is caused mainly through the production of debris; thus, the changes in weight factors with beating cannot be correlated consistantly with freeness.

When a fiber splits lengthwise the weight factor of that fiber is halved. As the weight factor varies directly with the average weight per unit length of the fiber, which is measured by the International denier or grex, accurate weight

factors for pulps, especially when they are well beaten, cannot be determined until an agreement is reached as to the minimum width of fiber elements which are to be included in weight factor determinations.

The choice of rag as a basis for the determination of weight factors was an unfortunate choice for two reasons. (5) First, rag fibers split lengthwise in beating, and second, the nature of the rag fibers in a furnish is very indefinite.

Wood and grass fibers have little tendency to split lengthwise in beating, splitting only with difficulty. When the weight factor of a wood or grass fiber is determined using unbeaten rag fibers as a base, the weight factors of the wood or grass fibers progressively decrease with the degree of beating. When wood and rag fibers are beaten together, the weight factor of the wood fiber increases with the degree of beating. Table II prepared by Mr. Clark compares the weight factors, which were determined by the grex and by the width of the fibers, with the weight factors determined by Mr. Graff.

When cotton linters are used in a paper furnish instead of staple cotton fibers, the weight factor of one which is used for cotton rag fibers, is too low. The weight factor of virgin cotton linters decreases with the degree of freeness

until a freeness of 405 is reached, where the linters have a weight factor of one which is the same as the factor for cotton rag stock. (10) Therefore, the source of the cotton fiber in a sample is of considerable importance along with the observed effect of the freeness of the sample. Second cut cotton linters have the highest weight factor, followed in order by first cut cotton linters, and staple cotton. If these factors are to be taken into consideration, the nature of the source of the cotton in a furnish must be known or determined. Table III shows the change in the weight factors of first and second cut cotton linters with the change in freeness.

Cotton linters are thick walled and difficult to hydrate. It is possible to differentiate between long staple cotton and cotton linters by staining with a combination of an orange and blue dye. The dye is made up of a direct orange dye with a color index number of 621 and a direct blue dye of color index number 518. Pontamine Sky blue 6 B X Greenish, and Pontamine Fast Orange 6 R N Concentrated have the correct color indexes. The dye, which was developed to determine the degree of beating , is made up of a one percent solution of each. Equal parts are mixed together for use on wood or grass stock. For use on rag stock forty-five parts of the

blue solution are mixed with fifty-five parts of the orange solution. When this dye is used on a mixture of cotton linters and staple cotton, the cotton linters stain a deeper blue than does the staple cotton. The only orange dye which the linters will take up is in the fully separated fibrils. (20)

STAINING REACTIONS

One method of identifying the fibers in a sample is to use a stain that will give the fibers a deffinate color, which is not matched by any other fiber with the same dye or stain. Most of the stains which are used in fiber identification are of the iodine-iodide-metallic salt type. The staining of fibers by these stains is a colloidal phenomenon. The color reaction given by these stains is influenced by any condition or substance which may change the nature of the iodine adsorbed by the fibers. (19) Ligneous materials always absorb iodine to give a yellow color, which is produced by the uniform adsorption of small particles of iodine on the unhydrated lignin. The yellow color is not affected by any of the metallic salts used in iodine stains, and neither water or potassium iodide affect the staining to any great degree. The staining of cellulose apparently consists of the adsorption of the iodine-iodide complex and water around the hydroxyl groups. The colors vary from orange to red,

to violet to blue, depending on the amount of the iodine - iodide complex that is adsorbed by the fibers. The amount of the complex that will be adsorbed by the fibers can be increased by hydrating and swelling the fiber and can be decreased by adding a dispersing salt or by heating. The potassium iodide is used to form the iodine-iodide complex in the stain. The metallic salts in these stains swell the fibers and increase the amount of iodine adsorbed on the fiber.

Cell walls which contain lignin can be quickly and reliably detected by staining with a colorless benzidine solution. Lignified cell walls are stained a yellow or orange color, but no color is developed in cellulose or in cutinized or in suberized cell walls. (17) This dye can be used as a method of determining groundwood pulp, but it does not differentiate between bleached and unbleached groundwood.

Well known stains such as phloroglucinol, aniline sulfate, the iodine-iodide-metallic salt stains (Herzberg and C stains), the W stain, and texchrome have been used in an attempt to find a means of determining whether groundwood pulp found in a paper furnish was bleached or unbleached. When these stains were used, no differences in the staining reactions could be discovered, even with the use of polarized or fluorescent light. (11)

Investigations made with the bright stain showed some promise. (12) Under ordinary light the bright stain would stain unbleached groundwood from a blue red to a red. When bright stain was tested under ~~a fluorescent reaction of~~ ultra-violet light, unbleached groundwood showed fluorescent colors ranging from a dark purple drab to a dark vinaceous gray. Under the same lighting conditions bleached groundwood gave a light vinaceous gray color. However, this staining method was not entirely satisfactory because in mixtures or with lightly bleached groundwood, the difference in color was uncertain.

The best results were obtained with the Lofton-Merritt stain. The Lofton-Merritt stain used in the tests was prepared by mixing twenty parts of a one percent fuchsin solution with ten parts of a two percent solution of Malachite green. Three parts of a five hundreths solution of hydrochloric acid was added to the dyes. The fuchsin used was General Dyestuff's Magenta AB powder. (11) The slides were prepared by adding ~~ten~~ two or three drops of the stain to the fibers, which had been dried on the slide at a temperature of sixty degrees centigrade. The stain was allowed to remain in contact with the fibers for two minutes at which time the excess stain was drained off and the fibers were washed with

distilled water. When the Loften-Merritt stain is used, the fibers may be stained in a beaker and the stained fibers dried on the slide. With this staining method the unbleached groundwood stained from a deep blue violet to a pale violet color. Commercially peroxide bleached groundwood gave an amethyst violet to a light hortense violet color. When known mixtures of bleached and unbleached groundwood were analyzed with this staining method, results showed a standard deviation, which was entirely consistent with accurate fiber analysis, and indicated that results can be obtained with a small percentage of error.

In all staining operations the color which is obtained with any particular stain will vary according to the light reflected into the microscope and on the extent of cooking, bleaching, and refining which the pulp has undergone. (8) Care should be taken in preparing the stains which are to be used to stain fibers as varying results will be obtained if the stains are not correctly prepared. (21)

The use of stains to differentiate between hardwood pulps cooked by the soda, kraft, or semichemical processes is very unreliable. There are some cases in which these pulps can be distinguished, but in most of the attempts to separate these fibers by staining methods the results were

not sufficiently accurate for fiber analysis. (22) Pulps processed by a full kraft cook or a semichemical kraft can be separated by staining. The semichemical pulp gives a deeper lignin color than the full kraft cooked pulp does.

Acid or alkaline cooked pulps can be determined by the appearance of fine transverse splits or fractures which appear in the fiber tracheids and in some bast fibers after the fibers have been treated with a boiling ten percent solution of sulfuric acid followed by swelling in a fifteen percent solution of sodium hydroxide. (6) The fractures appear only in the acid cooked pulps or in alkaline cooked pulps which are prepared from wood that has been subjected to acid hydrolysis before pulping. The number of fractures which appear in the fibers increases with the severity of the acid cook, and with the length of time that the fibers are treated with the sulfuric acid solution. With careful control it may be possible to use this method to determine if a semichemical pulp has been prepared by a kraft cook or a neutral sulfite cook.

FIBER CHARACTERISTICS

Staining reactions are, of course a great help to the microscopist in the identification of fibers, but it cannot be toomuch emphasised that the real criterion for the identification of fibers is a knowledge of their structure. (1)

The paper making process may alter the structure of the fibers especially, if they have undergone prolonged beating. After prolonged beating with the possible exception of Norway pine, identification of the wood species is practically impossible. (3) During the examination of fibers it is a good practice to follow a routine method. (2) The use of polarized light in the examination of fibers often aids the microscopist in the identification of otherwise hidden characteristics.

Of the fibers discussed in this article only West coast coniferous kraft and cotton linters can be identified by the fiber structure. Pulp woods used in the production of semi-chemical pulp, hardwood kraft, and hardwood sulfite are also used in soda pulp.

Western kraft pulp is made from douglas fir, western hemlock, western red ceder, and loblolly pine. Unbroken fibers of western hemlock range in length from 1.3 to 6.3 millimeters, with an average length of 4.2 millimeters. Douglas fir fibers range in length from 1.7 to 7.0 millimeters, with an average length of 3.9 millimeters. The average width of western hemlock fibers is between thirty and forty microns, and the average width of douglas fir fibers is between thirty-five and forty-five microns. (13) Douglas fir also has spiral thickening on the tracheid walls. Table IV gives the charac-

teristics of unbleached western krsft fibers.

First cut cotton linters are produced when from twenty to seventy-five pounds of linters per ton of seed is produced. The second cut linters produce from one hundred to one hundred eighty pounds more of linters. (14) In order to swell the fibers so as to reveal their microscopic details a dilute solution of cuprammonium hydroxide is used. The characteristics which distinguish cotton linters from staple cotton are not sharp. In general linters fibers are coarser, darker, and shorter that staple fibers. Linters fibers rarely exceed a few eighths of an inch in length. They are as a rule slightly greater in diameter than staple fibers, ranging in diameter from fifteen to twenty microns. The average fiber length is 3.1 millimeters for first cut linters and 2.3 millimeters for second cut linters. Linters are more nearly cylindrical than staple fibers, and they have thicker walls and narrower central canals.

CONCLUSION

The microscopist is in the same position today that he was when the C stain was first developed. There are so many new fibers ~~that~~ which present day stains will not identify that a new stain is needed.

TABLE I (13)

Weight Factors of New Pulps

Pulps	Weight Factor	Standard Deviation
Semi-Chemical		
Spruce (unrefined)	2.10	0.54
Spruce (peeled)	1.69	0.32
Average pine and spruce	1.91	0.49
Gumwood (unknown)	1.38	0.14
Gumwood (neutral sulfite)	1.25	0.09
Gumwood (Average)	1.32	0.14
Cotton linters		
Freeness 751	1.51	0.07
Freeness 710	1.30	0.06
Freeness 405	1.02	0.16
Chemical pulps		
Black gum (soda)	1.06	0.12
Red Gum (soda)	1.17	0.16
Tupelo Gum (soda)	0.91	0.16
Average for gumwood soda	1.06	0.14
Red Gum (kraft)	0.97	0.08
Red Gum (kraft refined)	1.03	0.17
Average gumwood kraft	0.99	0.14
Average gumwood chemical	1.03	0.17

TABLE II

Comparison of Weight Factors (5)

Fiber	Grex gm/10^3 M	Weight Factor from Grex	Weight Factor from width	Average Width Microns	Weight Factor Graff
Rag	1.8	1.00	1.00	20	1.00
Cotton Linters	——	----	1.5	30	1.5
Deciduous					
Sulphate	- - -	----	----	----	0.7
Sulphite	1.2	0.65	----	----	0.6
Poplar	——	----	0.6	22	0.55
Groundwood	---	----	1.3	40	1.3
Coniferous					
Southern Pine	1.6 or 2.7	0.9 or 1.5	1.05 or 1.55	32 - 46	1.55
Western Hemlock	2.45	1.3	0.95 or 1.15	28 - 35	1.2

TABLE III (15)

Weight Factors and Freeness of Cotton Linters

Sample number	Freeness (Schopper Reiger)	Weight Factor
	Second Cut Linters	
I-a	Unbeaten - 885	1.91
II-a	Unbeaten - 885	1.92
1	815	1.46
2	785	1.45
3	750	1.52
4	725	1.36
5	600	1.29
6	510	1.10
7	435	1.06
	First Cut Linters	
I-a	Unbeaten - 870	1.56
II-a	Unbeaten - 870	1.72
1	805	1.34
2	695	1.19
3	595	1.10
4	560	1.02
5	490	1.00
6	475	1.02
7	380	0.95

TABLE IV

CHARACTERISTICS OF UNBLEACHED WEST COAST KRAFT FIBERS (13)

	Douglas Fir	Hemlock	Lodgepole Pine	Bl. Spruce	Red Cedar
Fiber Length, mm					
Arithmetical Avg.	2.62	2.21	2.54	2.14	2.05
Weighted Avg.	3.38	2.80	3.17	2.86	2.76
Fiber Width, mm	0.044	0.041	0.043	0.037	0.040
Area, Sq. mm	0.112	0.100	0.113	0.084	0.089
Fibers / Gm., millions	3.5	4.5	3.8	4.3	4.5

BIBLIOGRAPHY

(1) Armitage, F. D. The Microscope and Its Applications in the Papermaking and Printing Industries. 4. The preparation of Samples for Examination. Paper Making 67, no. 1:21-24 (Date)

(2) Armitage, F. D. The Microscope and Its Application in the Papermaking and Printing Industries. 5. An Atlas of Papermaking Fibers. Paper Making 67, no. 1:58-59, 61

(3) Alliot, H. Microscopical Examination of Wood Fibers During the Beating or Pressing of Paper Pulps. Bulletin of the Technical Com. Pap. Franc., 1946:325-328; Chemical Abstract 42:2768

(4) Berke, Ralph. Fiber Length Measurements. Chemist Analyst. 28, no. 3:57, 59 (November, 1939)

(5) Clark, James d'A. Notes on Weight Factors for Fiber Microscopy. Tappi 34, no. 7:317-318 (July, 1951)

(6) Dolmetsch, H., Franz, E., and Cofrens, E. Influence of the Morphological Structure of Pulps upon Their Reactions. Journal of Makromal Chemistry. 1, no. 7-9:167-184 (June, 1944) Bulletin of the Institute of Paper Chemistry 16:135

(7) Fyfe, Thomas. An Optical Projection Method for Measuring Fiber Length. Tappi 34, no. 7:306-310 (July, 1951)

(8) Graff, John H. New Stains and Their Use for Fiber Identification. Paper Trade Journal, 100, no. 16:45-50 (April 18, 1935)

(9) Graff, John H. Pulp and Paper Microscopy. Appleton, Wisconsin. The Institute of Paper Chemistry, 1942.

(10) Graff, John H. Additional Weight Factors of Pulps. Technical Association Papers 27, Pages 471-472

(11) Graff, John H. Stain Reactions of Unbleached and Bleached Groundwood Pulps. Paper Trade Journal 118, no. 16:29 (April 20, 1944)

(12) Graff, John H. Remarks on C Stain in Connection with Groundwood Bleached with Peroxide. Paper Trade Journal 119, no. 24:32

(13) Graff John H. and Isenberg, Irving H. The Characteristics of Unbleached Kraft Pulps from Western Hemlock, Douglas Fir, Western Cedar, Loblolly Pine, and Black Spruce. II. The Morphological Characteristics of the Pulp Fibers. Tappi 33, no. 2:94-95 (February, 1950)

(14) Hock, Charles W. Structure of Cotton Linters. Textile Research Journal. 17, no. 8:423-430 (August, 1947)

(15) Isenbefg, Irving H, and Peckhan, Charles L. Weight Factors for Cotton Linters. Tappi 33, no. 10:527-528 (October, 1950)

(16) Kupachka, B. Francis. A Universal Scheme for Wood Identification. Tappi 33, no. 9:66A, 68A (September, 1950)

(17) Lominski, Iwo, R. W., and Hutchison, S. A. A Simple Technique for the Differential Staining of Lignified Cell Walls. Nature 162, no. 4115:415 (September 11, 1946)

(18) Mason, S. G. The Specific Surface of Fibers--Its Measurement and Application. Pulp and Paper Magazine of Canada 51, no. 9:109-115 (August, 1950)

(19) Rowe, Herbert W. The Nature of Fiber Staining by Iodine Stains. Paper Trade Journal 116, no. 10:26-34 (March 11, 1943)

(20) Simons, Francis L. A Stain for Use in the Microscopy of Beaten Fibers. Tappi 33, no. 7:312-314 (July, 1950)

(21) Strachan, James. Chlor-Zinc-Iodine Stain. Paper Maker 112, no. 2:TS9 (August, 1946); Bulletin of the Institute of Paper Chemistry 17:262

(22) Sitterson, C. B. Letter to author from the Institute of Paper Chemistry. November 23, 1951

OUTLINE OF PROPOSED EXPERIMENTS

OBJECTive

To determine the effect that beating time has on the weight factors and the length and width of fibers.

PROCEDURE

1. Make beating run on staple cotton fibers, taking samples at beating times of 1, 3, 5, 10, 15, 30, 45, 60, and 75 minutes.
   a. Take Freeness on each sample
   b. Determine average fiber length and width
   c. Determine weight factor using sulfite fibers which are compared with the unbeated sample, as a standard.
   d. Determine if th orange-blue dye described in thesis differentiat between unbeaten and beaten fibers. If it will, determine weight factor against unbeaten rag.
2. Repeat step 1 using different weights on beater arm.
3. Repeat ste 1 and 2 with cotton linters.
4. If pulps and time are available repeat procedures 1 and 2 with all of the fibers called newely developed fibers in the literature survey of the thesis.
5. If microtome is available, make cross sections of all samples and determine the cross sectional area and grex of each sample.

EQUIPMENT

Microscope	Slides
Microprojector	Coverglasses
Microtome	Beater

Materials and Reagents

Herzberg Stain — C Stain

Pontamine Sky Blue 6BX Greenish dye — Color Index No. 518

Pontamine Fast Orange 6RN Concentrated — Color Index No. 621

Fuchsin -- General Dyestuff's Magenta AB powder

Malachite Green dye

Pulps

- Staple Cotton rag
- Cotton linters
- Douglas Fir Kraft
- Western Hemlock Kraft
- Hardwood Kraft
- Neutral Sulfite Semi-chemical
- Kraft Semi-chemical
- Bleached Groundwood
- Unbleached Groundwood

EXPERIMENTAL INVESTIGATION

The experimental work which was carried out was directed toward finding a means of differentially staining hardwood pulps cooked by the soda process, kraft process, and neutral sulphite semichemical process.

It has been proven that the staining reactions of pulps with the iodine--iodide stains is the result of the way and the amount of the iodine--iodide complex that is adsorbed on the surface of the fiber. As the three above mentioned pulps are all cooked by a different process, it would seem probable that the cellulose structure of each pulp should be modified in a different manner and that the iodine--iodide complex would be adsorbed in a different manner on each.

When Graff's "C" stain alone is added to bleached fibers cooked by the kraft and the soda process, there is a very slight difference in the intensity of the blue color that is developed. This difference is so slight that it is very difficult for even a trained and experienced microscopist to tell the difference between the two fibers.

It is also a known fact that different fibers will dye differently with the same dye. Would it be possible to dye the fibers with some dye or a mixture of dyes, altering the surface characteristics of the fibers so treated.

Then when "C" stain, Herzberg stain, or some other dye or stain is added to the fibers, any slight difference in color reaction might be magnified, making identification of the fibers possible.

It was along this line that the experimental work was conducted. It could also be possible that different types of reflected light would show different color characteristics. Therefore, each prepared slide was inspected, not only under daylight, but also under ultraviolet and fluorescent lights. Early in the experimental work a small difference was discovered in the color reactions of all three pulps under the daylight rays. Under fluorescent and ultra violet light the lifference in colors was either non-existant or not as intence; therefore, examination of the prepared slides under ultra violet and fluorescent light was stopped, and all slides prepared after this were examined under daylight only.

The fibers used in the experimental work were bleached hardwood soda and bleached hardwood kraft from the International Paper Company, bleached and unbleached semichemical neutral pulps from the Watervliet Paper Company, and unbleached neutral sulphite pulp from the Otsego Falls Paper Company.

The first slides prepared were stained with "C" stain.

The resulting colors for the bleached fibers were a medium blue. The vessels were a lighter blue than the fibers, and the kraft fibers were possibly a little darker than the soda fibers; with theneutral sulphite semichemical fibers being both the light and the dark shades of blue. Under the rays from the fluorescent light the same colors were obtained, as under daylight which was provided by a scopelight. Under the ultra violet light the vessels were a medium violet, and the fibers were a dark purple black. All three pulps showed the same color reactions under the ultra violet light.

The unbleached neutral sulphite semichemical vessels stained a light yellow and the fibers were yellow in the center, turning green toward the outside edges. Some of the fibers were entirely green. The unbleached semichemical fibers with a higher lignin content stained a deeper yellow, almost an orange.

In staining these slides "C" stain was placed on the slides and allowed to remain in contact with the fibers for one minute, three minutes, and six minutes. At the end of this time the excess stain was squeezed from the slide with the cover glass. The length of time with which the stain was in contact with the fibers did not seem to make a noticeable difference in the colors developed.

The slides were also examined at intervals of fifteen minutes, thirty minutes, one hour, and two hours after treatment with the stain. At the end of two hours time the unbleached slides developed the same color of blue as the bleached slides.

Slides were also stained with Herzberg stain; the slides being prepared in the same manner as those prepared with "C" stain. With daylight the bleached vessels were a light blue to a lavender, and the fibers stained from a dark purple black to a black. The kraft fibers were again slightly darker than the soda fibers. The difference in shade between the two fibers was not distinct enough to make a definite and positive identification. Under ultra violet light the colors of the fibers had a reddish cast.

With the Lofton-Merritt stain both the vessels and the fibers of the bleached pulps were stained a medium blue under the light from the scopelight and the fluorescent light. Under the rays of the ultra violet light the bleached pulps were a lavender to a purple color.

As none of the slides prepared in this manner showed any distinguishing color differences, "C" stain was added to the slides already prepared with Herzberg stain and with the Lofton-Merritt stain. No color differences could be

noted on the slides on which both Herzberg stain and "C" stain were used.

Considerable difficulty was encountered when an attempt was made to stain the slides with the Loften-Merritt stain followed with "C" stain. If the Loften-Merritt stain was not allowed to stay on the fibers for at least two minutes all of the fibers were not stained evenly. If the dye was allowed to remain on the fibers for period than for two minutes, no change in the staining reaction could be determined; therefore, it was decided that the optimum staining time for the Loften-Merritt stain was two minutes.

It was also difficult to obtain a slide in which the fibers were clear and distinct. When the excess Loften-Merritt stain was forced from the slide with the cover glass or blotted off with a blotter, dark black spots remained on the slide, which covered up the fibers and the colors obtained when the "C" stain was added.

Several slides were prepared in this manner and on the slides in which the black specks, probably undissolved dye particles, did not appear different shades were obtained for the three bleached pulps. The soda fibers stained a grey green, the kraft fibers a blue to blue black, and the semichemical fibers stained a darker grass green. Under the ultra violet light these fibers were all the same colors.

Mixtures were prepared of two of the bleached fibers, kraft and soda, kraft and neutral sulphite, and soda and neutral sulphite. A mixture was also prepared containing the three fibers. Slides were prepared of each of these mixtures, and an attempt was made to count the fibers on the slides to see if accurate fiber counts could be made. These attempts to count fibers were not successful for two reasons. First--the dark spots on the slides made it very difficult to even count the fibers and impossible to accurately differentiate between the colors formed. Second--from week to week the shades obtained and the degree of difference in color changed.

In an effort to overcome these difficulties the excess Lofton-Merritt stain was washed from the slides with distilled water before the "C" stain was applied. On these slides very little color difference was obtained.

Because of the difficulties encountered in using the Lofton-Merritt stain, an attempt was made to find some other dye or stain that would alter the surface characteristics of the fibers being tested. With this in mind over thirty different dyes were used, singly and in pairs. No promising results were found in this series of tests.

When the excess dye was blotted from the slide before the "C" stain was applied, a dirty blotched color was the result. Washing the excess dye from the slide with distilled water was not the answer. When the excess dye was washed from the slide, most of the dye was also washed from the fibers when the excess dye was washed from the slide. The method finally used with the most success was to place seven or eight drops of a two and one half percent alum solution on the slide. To the alum two or three drops of the dye being used were added. This was allowed to set for two minutes. Then, the excess dye-alum solution was drained from the slide; the slide was washed with distilled water, blotted dry, and the "C" stain applied.

Using this method, the dye which gave the most promising results was a one percent solution of Fuchsin. This combination of dye and stain gave a very different and distinct color to each of the three pulps being tested. The color of the soda fibers ranged from various shades of a light blue or blue grey to a light blue shaded with brown. The color of the kraft fibers was a dark blue to a blue black. The semichemical fibers were various shades of green.

Armed with these encouraging results, slides were prepared from a mixture of equal part s of the three bleached

pulps. The fiber counts, which were made on the first eight of these slides, agreed within a tolerance of plus or minus five percent. To stain the next slide a new bottle of freshly prepared "C" stain, purchased from The Institute of Paper Chemistry, was used. Fiber counts were impossible on the resulting slide, and on all other slides on which this new bottle of stain was used. Slides were then prepared ofeach separate kind of fiber and stained with the fresh stain. All of the fibers were about the same color.

As the shades obtained from week to week had varied, a small portion of the "C" stain was allowed to age for one week in a stoppered clear glass test tube. At the end of the weeks time, slides were made and stained with the aged "C" stain. The colors obtained were about the same as those obtained with the old bottle of "C" stain before the new stain was opened.

CONCLUSION

In the experimental work carried out during the past semester some promising results have been obtained. It seems that it may be possible through the use of the method described to differentially stain bleached kraft, bleached soda, and bleached semechemical pulps. There is still a large amount of work to be done before the process outlined

can be considered to be accurate and precise. Only one pulp of each different cook was used. Is the staining method used adaptable to all soda, kraft, and semichemical pulps or will the degree of delignification change the results? The "C" stain is another variable that will have to be definitely determined. The "C" stain used to obtain results was either artificially aged in clear glass or aged by time in a dark bottle. Can the needed stain be produced by using a smaller concentration of the iodine--iodide solution used in preparing the stain? These questions must still be answered, and many slides will have to be prepared and examined before the staining method described can either be accepted or rejected completely.