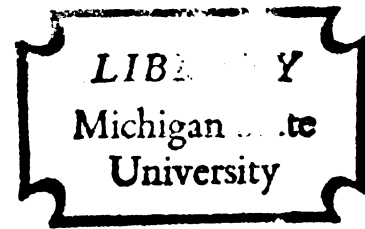


THE DEVELOPMENT OF A PROCEDURE TO ANALYZE
THE COMPOSITION OF CORRUGATED LINERBOARDS

Thesis for the Degree of M. S.
MICHIGAN STATE UNIVERSITY
CHARLES ROBERT VanHUYSEN, JR.
1972



LIBRARY

Michigan State
University

L

MICHIGAN STATE UNIVERSITY LIBRARIES



3 1293 00870 9564

092

OCT 2 1973

609

1

ABSTRACT

THE DEVELOPMENT OF A PROCEDURE TO ANALYZE THE COMPOSITION OF CORRUGATED LINERBOARDS

By

Charles Robert VanHuysen, Jr.

A procedure to determine the composition of corrugated board liners was developed as a beginning for research into recycled fiber usage. No existing research was found to use as a starting point. The procedure was based on screening to separate the sample by fiber length and a microscopic analysis to determine the composition of each fraction. Some physical testing was included as a basis for rough comparisons. The anticipated ultimate result of the application of the procedure will be a technique to determine the percentage of recycled fiber in a corrugated linerboard. The theory behind that result is that multiple refinings of a particular fraction within the sample will produce a predictable average reduction in average fiber length.

THE DEVELOPMENT OF A PROCEDURE TO
ANALYZE THE COMPOSITION OF
CORRUGATED LINERBOARDS

By

Charles Robert VanHuysen, Jr.

A THESIS

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTERS OF SCIENCE

School of Packaging

1972

ACKNOWLEDGMENTS

I would like to thank Dr. Wayne H. Clifford and Dr. James W. Goff for their direction during my graduate work at Michigan State University.

I would also like to express my appreciation to my wife, Sue Ann, for her support during my graduate work and for her help in preparing this thesis.

TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS	ii
LIST OF TABLES	iv
LIST OF FIGURES	v
INTRODUCTION	1
 Chapter	
I. ANALYSIS OF THE PROBLEM	3
II. ANALYTICAL PROCEDURE	8
Sample Disintegration	8
Screening Procedure	11
Preparation and Analysis of Microscope Slides	12
Physical Testing Procedure	15
III. DISCUSSION OF TESTING PROCEDURES	17
Discussion of the Disintegration Procedure	17
Discussion of the Screening Procedure	20
Discussion of Microscopic Analysis and Slide Preparation	22
Discussion of Physical Testing	23
IV. SAMPLE MICROSCOPIC ANALYSIS	26
V. SUMMARY AND CONCLUSIONS	30
APPENDIX	32
Wood Structure	32
Species Identification	34
LIST OF REFERENCES	40

LIST OF TABLES

Table		Page
1.	Mesh Size of Screens in 10" Diameter Sieves . .	12
2.	Colors Developed by the C-Stain	14
3.	Colors Developed by the Wilson Stain	15
4.	Weight Retained on Each Sieve	26
5.	Ratio of Softwood to Hardwood on Each Sieve . .	26

LIST OF FIGURES

Figure		Page
1.	Mechanical Disintegration Apparatus	10
2.	Structure of Softwood Pit Types	36

INTRODUCTION

Paper manufacturers have been routinely including recycled fibers in corrugated linerboards for years. With the pressure to use recycled products, the government and others are specifying the percentage of recycled fiber required in the liners of the corrugated board they purchase. Meaningful specifications require some method of checking the resulting product. Techniques do not now exist to separate the recycled and the non-recycled fractions within a paper sample. Therefore, successful monitoring of these corrugated board specifications is impossible.

Specifications of the percentage of recycled fiber in a paper sample can be extremely deceiving. Recycled papers differ as a function of not only the level of recycling, but also the quality and type of the recycled fibers used. A recycled linerboard containing 20 percent Kraft roll trim is obviously different from one containing 20 percent recycled newsprint. Both linerboards could be lumped under the 20 percent recycled classification. To avoid this misleading labeling a classification system for recycled fibers on the basis of source, quality, and amount should be developed. The lack of research in this area is a major problem for such developments.

The problem was to design a method to determine the percentage of recycled fiber in a corrugated linerboard sample. No existing procedures were available to evaluate as a starting point. Consequently, the problem was simplified to designing a procedure that would lead to the ability to microscopically determine the amount of recycled fiber present. This procedure is necessary for continued research into the effect of recycled fibers on corrugated linerboards, because accurate comparisons between samples require carefully specified furnishes. The approach to solving the problem involved evaluating several alternative procedures with both a literature search and trial and error testing. The resulting procedure, its operation, and the theory behind it is described in the thesis.

CHAPTER I

ANALYSIS OF THE PROBLEM

Microscopically, a recycled fiber cannot be differentiated from a non-recycled fiber. The number of times an individual fiber has passed through a papermaking process cannot be detected by optically examining that fiber. In papermaking, the principle changes in the fiber structure occur in the beating or refining stage. Beating is the mechanical alteration of the fiber by the action of a stationary and several moving blades. It produces two primary effects on the fiber, transverse cutting and fibrillation or unraveling of the fiber components. The degree of each is dependent on the characteristics of the individual beater or refiner and the severity of the treatment. There are too many variables to allow the prediction of the appearance of an individual fiber after beating. Multiple beating cycles compound this difficulty. Individual recycled and non-recycled fibers cannot be separated without this predictive ability.

If the effect of refining on individual fibers is ignored a solution to the problem may be possible. Viewing the components of a sample as a total furnish

allows comparison of average effects on similar samples. By evaluating the effect of refinement on the total furnish the problem of predicting the appearance of individual fibers is eliminated. The amount of transverse cutting increases with the severity of the treatment; the average fiber length is decreased by longer, more severe, refinements. The recycled fraction of paper sheets has received multiple refinings, probably totaling a more severe treatment than similar non-recycled fractions. The overall fiber length of the recycled fraction should be less than an identical fraction (species, pulping, etc.) receiving only one refinement. Assuming similar fractions exist within different linerboards, a more severe refinement can be located by detecting a significant change in fiber length between those fractions. The final step is to determine what level of severity, on the average, indicates recycling.

The initial problem is to determine if similar fractions can be distinguished in commercial linerboards. If these do not exist comparisons cannot be made and this approach must be abandoned as a dead end. The problem for the thesis then, was to design a procedure to provide general information concerning the composition of commercial linerboards. If similarities do exist, a more elaborate procedure will be necessary to complete the analysis. An added result of the initial analysis will be an analyzed sample library that will allow the comparison

of samples supposedly containing the same percentage of recycled fiber. The analyst will be able to determine if a supposedly 40 percent recycled fiber is like other samples supposedly containing 40 percent recycled fiber.

The resulting procedure consisted of three major sections; a screening analysis, a microscopic analysis for species and processing types, and a small amount of physical testing. None of the procedures were as specific as possible because general information concerning liner-board composition was required.

The disintegrated sample was fractionated on the basis of fiber length using a sieve column. The purpose of the fractionation was to determine how fibers of the same type were distributed throughout the sieve column. Two variables are being examined, how accurately the sieve column separates the fractions and the percentage of a fiber type that will be retained in one sieve fraction. The accuracy of separation can easily be analyzed by microscopically examining the consistency of the fiber lengths within that fraction. If the percentage of a fiber type retained on a particular sieve varies from sample to sample an analysis of the change in fiber length of that fraction is possible. Using this screening procedure it would be possible to obtain a weighted average fiber length for the furnish. This was not done because it is a general measure that provides no specific information concerning the fiber lengths of the

different fiber types within the furnish. The fractionation of the sample can be increased by adding more sieves to the sieve column.

The microscopic analysis consists of techniques to determine pulping type, processing type (bleaching, etc.), and the species present. The purpose of this analysis is to determine if definite fractions within the sample are identifiable. If specific fiber groupings are present, they can be checked for fiber length changes. A correlation between the number of fiber types present and the level of recycling should exist. This could serve as a qualitative test for the presence of recycled fiber in a sample. This analysis can easily be made more quantitative. The number of each fiber type within a sieve fraction can be counted. This approach will be necessary for the procedure to determine the average fiber length change that indicates recycling. The analyses for the initial thesis procedure are basically qualitative. The analyst comments on the relative proportions of each fiber type in a sieve fraction, but no rigorous fiber counts are undertaken.

The physical testing was not included in the procedure to differentiate levels of recycling. It was included to provide a more common basis to compare the samples. Two boards that appear to have similar furnishes may produce markedly different results in physical testing. This could reflect very little on the amount of recycled

fiber in the sample. Physical testing is a non-specific method of evaluating the other variables in the sample, as well as fiber length and furnish content.

Compression testing is a common method of evaluating containers and corrugated board.

Compression strength of a corrugated box is of importance both as a partial indication of its warehouse stacking performance and as an overall measure of the quality of the fibreboard and conversion efficiency For purposes of quality control and material specification at earlier stages in box manufacture, however, the box plant and board mill desire meaningful test methods for evaluating the potential performance of liners, medium, and corrugated board.

McKee et al. (1961), in the article quoted above, described container compression resistance as depending on both edge crush resistance and flexural stiffness of the corrugated board. Articles by Buchanan (1968), McKee et al. (1962), and Ranger (1967), in turn, related modulus of elasticity of the liners to the flexural stiffness of the completed board. By including edge crush resistance and modulus of elasticity in the procedure, two levels of manufacturing variables are covered. The modulus of elasticity measures effects on the liners. This test is more relevant to the linerboard analysis than the edge crush, because it evaluates only linerboard properties. The edge crush evaluates variables of both the materials composition and its conversion to corrugated board. It is included to provide a common reference point, the board structure, for comparisons among different samples.

CHAPTER II

ANALYTICAL PROCEDURE

Sample Disintegration

Approximately one square foot of the corrugated board sample was soaked in water until the liners separated from the corrugating medium. The liners were placed in the room for 24 hours. The moisture content of the partially conditioned sample was determined on an original weight basis according to TAPPI standard T-412 OS-63. Two to three grams of the sample were weighed; the amounts of oven-dry (O.D.) fiber in the sample was computed by subtracting the weight of water in the sample from the total sample weight. The final sample was rewetted, separated into one-inch squares, and soaked in water for at least eight hours.

The one-inch squares were separated into smaller squares, one square centimeter in area. These pieces were boiled in 500 milliliters (ml.) of a 5 percent aluminum chloride solution for from five to twenty minutes according to the expected amount of wet strength present. Before boiling, .05 gram of Ferric Oxide was added to the aluminum chloride solution. After boiling, the sample was

washed thoroughly with water on a number 100 sieve-- opening .150 millimeters. It was then brought to boil in 500 ml. of a .5 percent sodium hydroxide solution and thoroughly washed with water on a number 100 sieve. If the resulting pieces did not defiber easily when rolled between the fingers another attempt on a fresh sample with an alternative procedure was necessary.

The two alternatives to the preceding procedure are:

1. A 15 minute soaking in acetone is substituted for the aluminum chloride boil. The .05 gram of ferric oxide is added to the sodium hydroxide boil.

2. Boiling in 500 ml. of water containing a few drops of a concentrated acid is substituted for the aluminum chloride boil. The .05 gram of ferric oxide is added to the sodium hydroxide boil.

After the sodium hydroxide boil, the sample was soaked for two minutes in 200 ml. of .05 normal hydrochloric acid. It was then thoroughly washed with water on a number 100 sieve. The sample was placed in the disintegration apparatus (Figure 1), diluted to 1000 ml., brought to a boil, and subjected to 30 minutes of treatment at 2500 revolutions per minute--a total of 75,000 revolutions. The suspension was drained from the disintegrator. Any large non-defibered clumps were rolled between the fingers and added to the suspension. The container holding the

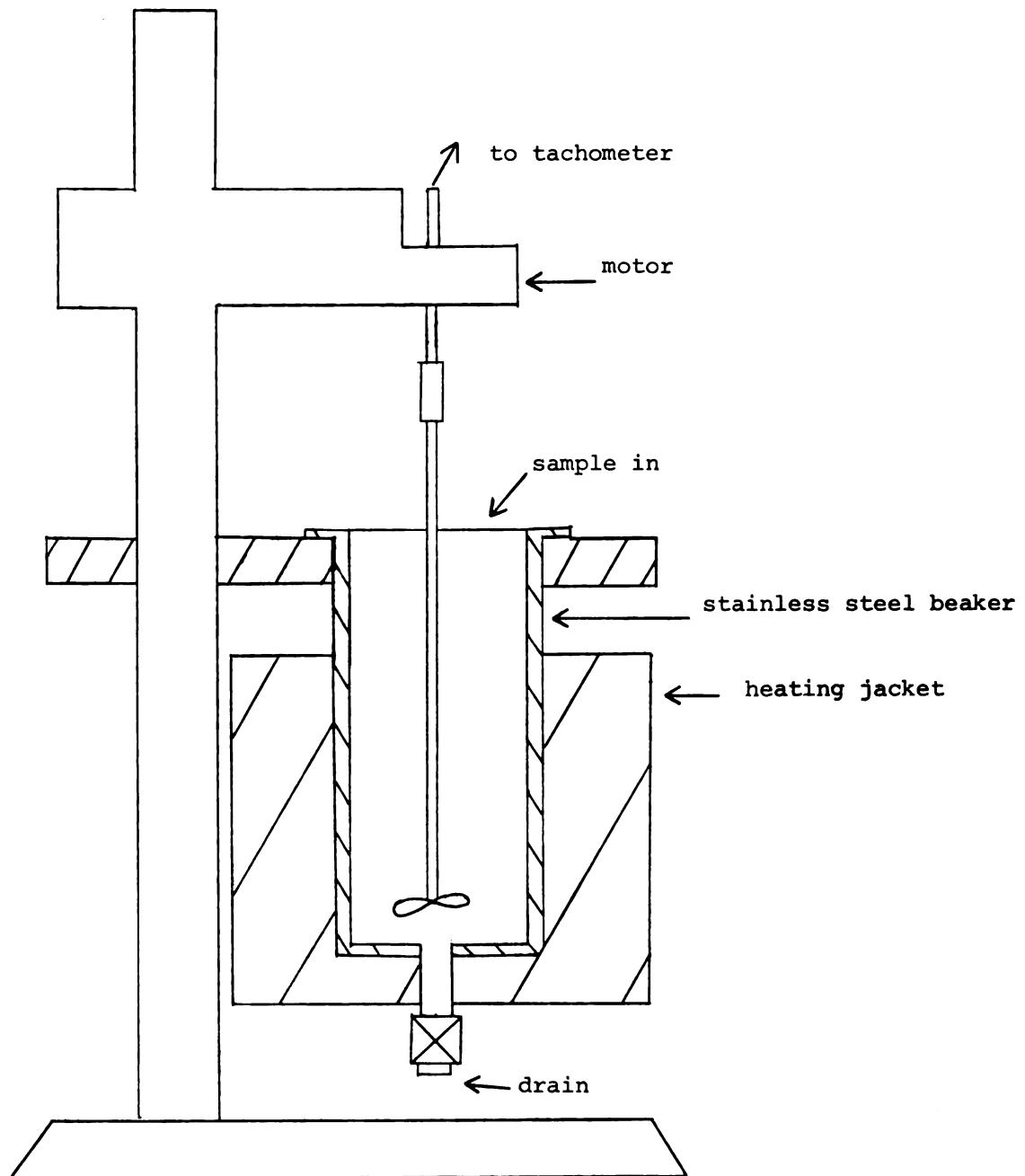


Figure 1.--Mechanical Disintegration Apparatus.

suspension was then shaken violently to induce fiber separation as a final preparation for the screening process.

Screening Procedure

A constant flow of water at 70° F and a flow rate of three gallons per minute was maintained through the sieve column. The water level was adjusted to coincide with the top of the sieve column. The joints between the sieves required heavy greasing to prevent leakage. The disintegrated sample was poured into the sieve column at convenient intervals, that maintained the water level. The water flow was continued for 20 minutes after the addition of the entire sample.

After draining the sieve column, (Table 1) the fibers remaining on each screen were washed into the Buchner suction apparatus and trapped on a filter paper of known dry weight. The pads were dried in an oven at $105 \pm 2^{\circ}\text{C}$ for eight hours. The weight of O.D. fibers from a particular sieve was calculated by subtracting the dried filter paper weight from the total weight of the dried pad. The ratio of the O.D. fiber weight trapped by each sieve to the total O.D. weight of the sample entering the sieve column was reported as the result of the screening. The percentage of initial sample weight not trapped on any screen was reported as having passed through the number 50 sieve.

TABLE 1.--Mesh Size of Screens in 10" Diameter Sieves.

Sieve Number	Opening in Millimeters
16	1.19
30	.595
50	.297

Preparation and Analysis of Microscope Slides

Approximately .01 gram of O.D. fiber from each sieve in the drained sieve column was placed into a separate 30 ml. test tube, suspended in 15 ml. of water, and the test tube was shaken to disperse the fibers. A glass marking pencil was used to draw two lines, one inch from each end, on clean dry microscope slides. The fibers were transferred from the test tube to the slide using glass tubing with an internal diameter of eight millimeters. The fibers were deposited at either end of the slide; two slides were prepared from each sieve. A slide was prepared from the sample before screening to facilitate hardwood species analysis. The slides were dried on a hot plate at 60°C. During the drying process the slide was tapped several times with a needle to disperse the fibers.

Two stains were employed for the microscopic analysis, Graff's C-stain and the Wilson stain. Two of the four fiber areas from each sieve fraction were stained using C-stain; the others were stained using Wilson stain.

Two to three drops of C-stain were applied to each fiber area, the fiber area was covered with a cover glass, and the excess stain was drained onto a blotter after exactly two minutes. A few drops of Wilson stain are placed on the fiber areas, covered with a cover glass, allowed to stand for ten seconds, and the excess stain is removed with a blotter. The colors developed by the C-stain are listed in Table 2; the colors developed by the Wilson stained are listed in Table 3. The two stains were used primarily to determine the type of pulping and processing the fibers had received.

The microscope was adjusted to Kohler illumination and a qualitative species analysis was performed using Panshin and DeZeeuw's (1964) key for woody fibers and TAPPI T10 SM-41 for non-woody fibers (see the Appendix for an explanation of their use). A qualitative analysis was also performed to determine pulping and processing type. The analysis was performed by sweeping the slide at two millimeter intervals, until the entire slide was covered, and recording every different fiber type that was encountered. The result of the qualitative analysis was a list of the species and, pulp and processing types present.

A partial quantitative analysis was then performed. An eyepiece with a pointer was added to the microscope and the 5 mm. sweeping procedure was again employed to evaluate the slide. As each fiber passed the pointer it

TABLE 2.--Colors Developed by the C-Stain (Isenberg, 1958).

A. C Stain

1. Groundwood: Vivid yellowish orange
2. Softwood pulps
 - a. Sulfite
 - (1) Raw: Vivid yellow
 - (2) Medium cooked: Light greenish yellow
 - (3) Well cooked: Pinkish gray
 - (4) Bleached: Light purplish gray to weak red purple
 - b. High alpha
 - (1) Unbleached: Very pale brown to brownish gray
 - (2) Bleached: Moderate reddish orange to dusky red
 - c. Sulfate
 - (1) Raw: Weak greenish yellow
 - (2) Medium and well cooked: Strong yellowish brown to moderate yellowish green and dark greenish gray
 - (3) Bleached: Dark bluish gray to dusky purple
3. Hardwood pulps
 - a. Sulfite
 - (1) Unbleached: Pale yellow green
 - (2) Bleached: Weak purplish blue to light purplish gray
 - b. High alpha
 - (1) Bleached: Moderate reddish orange to dusky red
 - c. Soda and sulfate
 - (1) Unbleached: Weak blue green to dusky blue green and dark reddish gray
 - (2) Bleached: Dusky blue to dusky purple
4. Rag: Moderate reddish orange
5. Manila
 - a. Raw: Light greenish yellow
 - b. Unbleached and bleached: Yellowish gray to weak blue and medium gray
6. Jute
 - a. Unbleached: Vivid yellowish orange
 - b. Bleached: Light yellow green
7. Straw, bamboo, cane, flax hurds, and esparto
 - a. Raw: Light yellow to weak greenish yellow
 - b. Unbleached and bleached: Light greenish gray to dark bluish gray and medium purplish gray
8. Japanese fibers
 - a. Gampi and mitsumata: Light greenish yellow to light bluish green
 - b. Kozo: Pinkish gray

TABLE 3.--Colors Developed the Wilson Stain (Isenberg, 1958).

Soda	Purple
Linen & Rag	Pink
Kraft	Brown (some gray)
Unbleached Sulfite	Colorless
Bleached Sulfite	Lavender
Bleached Sulfate	Blue
Cotton	Red
Groundwood	Yellow
Straw	Predominantly green

was recorded under the proper species and process type. The quantitative result of the species analysis was reported as the approximate percentages by number of softwoods, hardwoods, and non-woody species present in each sieve fraction. The quantitative result of the pulp and process type analysis was reported as the approximate percentages of the two most common types in each sieve fraction.

Physical Testing Procedure

The edgewise compressive strength test was run according to proposed TAPPI standard T 811. An Instron model TTC with a type E load cell served as the compression device. The test result was the average of five samples.

The same Instron using a type C tension load cell was used to run the stress-strain analyses. The liners were separated from the corrugated board samples by soaking in water. The liners were then conditioned for 48

hours at 70°F and 50 percent relative humidity. The sample was cut into strips, one inch wide and five inches long, in the machine direction. The analysis was run with these settings on the Instron:

Chart speed -- 20 inches/minute

Crosshead speed -- .5 inches/minute

Grip separation -- 4 inches

The modulus of elasticity was computed in pounds per square inch using this formula:

$$16 \times K01 \times K02 / (K05 \times K07) = E$$

K01 = load

K02 = Full scale load

K05 = Deformation

K07 = Sample cross sectional area

The result of the test was the average of five samples.

CHAPTER III

DISCUSSION OF TESTING PROCEDURES

Discussion of the Disintegration Procedure

The development of a procedure to disintegrate corrugated linerboard with the minimum of fiber damage proved complex. TAPPI 205 OS-71 describes a method for disintegrating pulp samples and Isenberg (1958) describes a method for disintegrating small paper samples for microscopic analysis. A technique for disintegrating large samples, minimizing fiber damage, was not available, consequently a procedure combining the two existing techniques was employed. The resulting procedure consisted of four basic steps:

1. Soaking in water
2. Removal of sizing and wet strength
3. Disintegration
4. Final preparation.

The most troublesome area of the procedure was removal of the sizing and wet strength agents.

Sizing a paper sheet and adding wet strength resins are different methods of producing water resistance within

a sheet. Sizing is added to prevent or delay water penetration; wet strength agents are added to preserve sheet strength once the material is wetted. Both processes are commonly used in corrugated linerboards. Sizing is often used to prevent excessive glue penetration into the liners. Rosin is the most commonly used sizing material. The amount retained in the sheet depends on the presence of aluminum salts, commonly alum, in the furnish. The precipitation of the rosin onto the fibers by the alum is easily blocked by the presence of ferric ions in the furnish. The introduction of a small amount of ferric oxide into the disintegration procedure during the aluminum chloride boil, promoted the removal of the sizing.

The most common wet strength resins are melamine-formaldehyde and urea-formaldehyde. Isenberg (1958) described a technique for removing these resins from samples to be used in microscopic analysis. It involved boiling the sample in 5 percent aluminum sulfate solution followed by boiling in .5 percent sodium hydroxide. The boiling in aluminum sulfate was replaced by boiling in an aluminum chloride solution which produces the same results. Procedures for the removal of other wet strength resins were also described by Isenberg. An acetone wash, substituted for the aluminum chloride boil, was designated to remove other resins--polyvinyl copolymer, polyvinyl chloride acetate, cellulose acetate butyral, chlorinated rubber, polyvinyl butyral, and ethyl cellulose. A final

approach is to replace the aluminum sulfate boiling with a boiling in water containing a few drops of concentrated acid. A simplistic test for the presence of wet strength resins involves rubbing the surface of the thoroughly wetted sample. If fibers release easily, wet strength effect is low. This test was used to check the sample for the efficiency of resin removal. If the wet strength resin was not removed by the initial treatment, the acetone treatment, first, or the concentrated acid treatment, second, was substituted. In most cases the treatment with aluminum salts is required, consequently the initial attempt at disintegration assumes the presence of melamine-formaldehyde or urea-formaldehyde resins. The second attempt, which is no more severe, is made using the acetone wash. The treatment using concentrated acid is a last resort, because of the degree of fiber damage it can produce. Concentrated acids attack both the lignin and cellulose fractions of the fiber, consequently, severe fiber degradation is possible.

TAPPI standard T-205 OS-71 describes a method for the standard disintegration of a pulp sample. It involves treatment of the pulp in an automatic stirring device at 3000 revolutions per minute for a total of 75,000 revolutions and at a consistency of 1.2 percent. The sample resulting from the wet strength and size removal procedures resembles a suspended pulp sample. The stirring apparatus for the thesis research (Figure 1) differed from the standard apparatus; the inside wall of the sample

container was smooth rather than baffled and the stirrer was run at 2500 revolutions per minute. Both of these differences led to a less severe treatment of the sample. The sample was suspended in 1000 milliliters of water and stirred with the suspension at a boil. The consistency was less than in the standard analysis. Running the disintegration with the suspension at boil could produce fiber damage if large residues of the solutions used to remove the size and wet strength remained in the sample. Consequently, the sample was washed extensively in both water and acid solutions, which served to neutralize the sodium hydroxide.

The final preparation for screening involved violently shaking the container holding the suspension to induce further dispersion.

Discussion of the Screening Procedure

The screening procedure separates the disintegrated fibers according to fiber length. Generally, fibers twice as long as the openings are trapped on a particular sieve. The separation is not absolute; fibers that should be trapped on a particular sieve are often passed through because of their orientation as they reach the screening surface. Consequently, long fibers are found on sieves with extremely small openings. Short fibers can be trapped on coarse sieves. As more long fibers become trapped on a sieve the opening size becomes smaller and fibers that

would normally pass through the sieve are trapped by the sieve-fiber network. If large samples are screened the pad build up effect is great, the coarser screens trap larger amounts of the sample than expected. The thesis research used a sample much smaller than that employed in standard analyses to eliminate the pad effect. The effect of poor fiber orientation could not be eliminated and was assumed to be the same for all screenings.

Fractionation of the sample is important because fiber length is an indication of the degree of refinement of the sample. The higher the level of refinement, the more of the sample will be trapped on the finer screens. The weight percentage trapped on the middle screens changes little as the switch from one level of refinement to another is made. The change in level of refinement is evidenced by the percentages trapped by the coarsest and finest screens.

It is possible to view the effect of change in degree of refinement on similar furnishes on two levels. The total furnishes can be examined by noting any differences in percentages by weight trapped on the different sieves. Particular species or pulp types common to each furnish can be compared by determining if a change in the trapping mesh size has resulted. This latter technique could lead to separation of recycled fractions.

The standard analysis, TAPPI T233 SU-64, is designed to determine a weighted average fiber length.

The thesis research is mainly looking for a fractionation of the sample. For simplicity, the number 100 sieve was eliminated from the standard sieve column (Table 1). Several preliminary screenings of disintegrated corrugated board liners produced no significant amounts trapped on the number 100 sieve.

Discussion of Microscopic Analysis and Slide Preparation

The slide preparation procedure was extracted directly from Isenberg (1958).

Fibers used in making paper are composed primarily of lignin, hemicelluloses, and cellulose. The amount of each component present depends on the type and degree of treatment that the fiber receives in the papermaking process. The color reactions produced by staining are, thus, dependent on the prior treatment of the sample. Pulped and bleached samples are low in lignin content, generally producing a bluish-red; groundwood is high in lignin content invariably producing a bright yellow. The analyst works in a continuum from yellow, high lignin content, to red, nearly pure cellulose.* Fibers receiving the same treatment do not always produce the same color reactions. Consequently, experience and corroborating evidence are important. The corroboration comes from other stains or from comparisons of color reactions with known standards.

*This assumes the use of C-stain or Wilson Stain.

Isenberg (1958) lists scores of stains that can be used in specific instances. For analysis of corrugated liner boards, C-stain (Table 2) and Wilson stain (Table 3) are most useful and produce the most overlap for confirmation. The standards are developed in the form of a library. Several of the differentiations are quite easy--bleached from unbleached and chemical from groundwood. The separation of the chemical pulps is difficult and not nearly as accurate.

The species analysis for woody fibers was completed using a key from Panshin and DeZeeuw (1964). The nonwoody fiber analysis was completed using TAPPI standard T-10 SM-41. The use of these keys is explained in Appendix I.

The qualitative analyses produced no significant difficulties. A complete quantitative analysis requires an expert with an extensive fiber standard library. The difficulties result from the non-specific color reactions and from the fiber fragmentation resulting, from the refining processes. To guarantee an accurate analysis, the quantitative analysis was simplified. Separating hardwoods, softwoods, and non-woody fibers is not difficult. Analysis of the pulp and process type involves only the two most common types in each fraction, making the analysis simpler.

Discussion of Physical Testing

The edge crush test was shown to correlate well with container compression performance by Jonson and Toroi

(1969). Theoretically, it approximates the vertical load, compression performance of the corrugated board near the edges of the container. The corrugated in those regions is restrained from buckling by the structure of the container. The edge crush is not an exact approximator, because a well defined sample is being tested; in case compression it is impossible to delineate an area as the area being tested. The result of the test is dependent on the sample size and preparation. Sample height influences the edge crush result by either promoting or limiting buckling. Generally, taller columns increase buckling and lower compression resistance. The edges of the sample must be cut cleanly and be parallel to promote even loading. The edge crush test described by TAPPI T811 differs from the Swedish Packaging Institute test in that the loading edges of the sample are paraffin coated. The typical failure of the Swedish sample is a roll down of the loading edge. This failure results mainly from the sample preparation, not from the compressive limit of the corrugated board. Paraffin coating of the edges eliminates this type of failure; the resulting failure is between the paraffin edges, remote from any of the vertical loading edges. The resulting edge crush values are significantly higher.

McKee et al. (1961) described total box compression strength as depending on both edgewise compressive strength and flexural stiffness of the corrugated board.

In their work edgewise compression is more important than flexural stiffness by a three to one ratio, but it is the flexural stiffness that causes the difference in compression resistance between A, B, and C flute corrugated board. The flexural stiffness approximates the bending characteristics of the unrestricted central portion of a corrugated container panel. The central portion of a corrugated container panel will generally deflect or bow before the total container fails, consequently flexural stiffness affects total container compression. The flexural stiffness of a corrugated board sample depends primarily on the modulus of elasticity of the liners and the thickness of the board. Modulus of elasticity is generally determined by calculating the slope of the initial straight line segment of a stress-strain curve. It is normally calculated from a tensile stress-strain curve. Setterholm and Gertje Jensen (1965) proved that the modulus of elasticity is almost exactly the same under tension or compression.

Edge compression tests are generally run on beam deflection type compression testers. The thesis uses an Instron load-cell type compression instrument, because of its availability and accuracy.

CHAPTER IV

SAMPLE MICROSCOPIC ANALYSIS

I. Screening: total weight of over-dry fiber = 2.260 grams

TABLE 4.--Weight Retained on Each Sieve.

Sieve	Weight of Oven-dry Fiber Retained	Percentage
16	1.600 grams	70.8
30	.605 grams	26.8
50	.040 grams	1.8
Fines	.015 grams	.7

II. Microscopic analysis:

A. Ratio of Softwoods to Hardwoods

TABLE 5.--Ratio of Softwood to Hardwood on Each Sieve.

Sieve	Ratio
16	7.0
30	2.5
50	0.9

B. Species

The softwood species present were Southern hard pine--pinoid pits--and a trace of soft pine--fenestriform pits. The hardwood species were unidentifiable because the vessel elements present were severely damaged.

C. Pulp and Processing Type

Most of the softwood fraction, approximately 90 percent of the total weight, was Kraft pulp. There were also traces of softwood groundwood present. The hardwood fraction was mainly bleached sulfate and totaled 8 to 10 percent of the sample weight.

III. Comments

The sample furnish was easy to analyze. It is almost entirely softwood Kraft, probably of the hard pine species. There are some hardwood fibers present; their species could not be determined because the corresponding vessel elements were destroyed. Seventy percent of the sample, by weight, was trapped on the coarsest sieve. The ratio of softwood to hardwood, seven to one, was somewhat misleading because it was determined by number. By weight, the ratio between the softwood and hardwood fractions would be much greater--possibly 10 or 15 to 1. Factors are available to correct for the weight differences, but in the initial general analysis it suffices to recognize the problem.

The sieve column appeared to produce an accurate separation. The coarser sieves and the long fibers did not appear to trap very many fibers that would normally pass through. This was checked while examining the microscope slides. The fibers in each fraction were of relatively uniform lengths. The highly damaged softwood fibers were located on the finest sieve, very few were found in the coarser fractions.

It was easy to speculate about the separate fractions within the sample. The sample appeared to be almost entirely hard pine Kraft, with small, probably recycled additions. The additions were mainly bleached hardwood sulfate stock. The species of these additions were indeterminate because only one vessel element was found intact. That appeared to be poplar, but no conclusions can be drawn from one vessel element. This analysis showed that the separations are possible. Continued analyses are required to determine if all furnishes are this easy and if more difficult furnishes can be analyzed. Not all furnishes will be based on only two fiber types.

Physical testing was included in the analytical procedure to provide a basis for comparison between similar furnishes. None of the samples analyzed in the development of the procedure were from samples similar enough to allow meaningful comparisons. Also, the form of the data from the physical testing is familiar. Consequently, the physical testing data was omitted. The microscopic analysis was

also designed to provide data for comparisons. Data from a sample microscopic analysis was included in the thesis to demonstrate the form and amount of information yielded by the procedure because the procedure is new.

CHAPTER V

SUMMARY AND CONCLUSIONS

The problem was to begin research to develop a technique for determining the percentage of recycled fiber in a corrugated linerboard sample. The approach was to develop a procedure to analyze linerboard samples based on fiber length screening, microscopic analysis, and some physical testing. The success or failure of the procedure depends in part on the ability to fractionate the sample accurately on the basis of fiber length and fiber characteristics--species, pulp type, and processing type. Consequently, the procedure is designed to provide general information to determine if these separations are feasible in commercial linerboards. It can be expanded to complete the development of the technique if the initial research proves feasible. More screens can be added to the sieve column to increase the number of fiber length fractions and the microscopic analysis can be made more quantitative. The steps were simplified for the initial procedure to reduce the time required for an analysis and to reduce the amount of information generated to what is initially important.

The procedure is a viable approach in the hands of an experienced analyst, without that experience it is worthless. A problem arises because the microscopic analysis of a sample is as much an art as a science. The differentiations in many cases are judgement calls, in which the odds on accuracy, without experience, are low.

Standard bases are necessary on which to compare the analyzed linerboards. One basis is included in the procedure, the edge crush resistance. Another is based on the samples to be analyzed. Analyzing linerboards that contain a specified recycled content will provide another basis for comparison. A library of boards at a specific percentage will be accumulated to use in analyzing other boards of that specification.

In conclusion, this procedure was developed as the beginning step into a relatively unresearched area. The analytical procedures are sound and will generate relevant information. This information, from many analyses, will form a bank of experience for the analyst. This experience may be the tool that allows the determination of the percentage of recycled fiber present. The procedure is designed to develop this experience, not to directly allow the separation of recycled and non-recycled fractions. The validity of the approach to the problem can only be proved or disproved through application of the procedure.

APPENDIX

Wood Structure

There are two principle directions in describing a block of wood. The radial direction is perpendicular to the growth ring and parallel to the wood ray. The tangential direction is perpendicular to the wood rays. There are two areas within a single growth ring, the earlywood and the latewood. Earlywood fibers are thin-walled with large lumens, hollow central portions; latewood fibers are thick-walled with narrow lumens. The latewood areas appear darker in a block of wood.

Longitudinal tracheids compose 90 percent of the volume of coniferous woods. Tracheids are relatively long, close ended cells that are oriented approximately parallel to the vertical axis of the tree. They provide mechanical strength and allow conduction of fluids. Most of the remaining wood volume of softwoods is composed of softwood rays which are perpendicular to the vertical axis of the tree. These rays are composed primarily of ray tracheids and ray parenchyma. Ray tracheids are like longitudinal tracheids in that they do not contain living contents (prosenchymatous). They also have bordered pitting. Parenchyma are short thin-walled cells with

simple pitting that contain living material (parenchymatous). These cells function in the storage and distribution of carbohydrates. The parenchyma and ray tracheids are important to species analysis because of the pitting that results when they cross a longitudinal tracheid. They are either altered or removed in the papermaking process to a degree that their structure is of little use in species analysis.

The structure of hardwoods is more complex than that of softwoods. The major prosenchymatous elements of hardwoods are vessel elements, tracheids, fiber tracheids, and libriform fibers. Vessel elements are the most important feature for hardwood species analysis. Their presence differentiates hardwoods from softwoods. Each vessel segment is a section of the pipe-like vessels, which serves a longitudinal conduction function. In the tree the hardwood tracheids are found surrounding the vessels. The papermaking process removes the hardwood tracheids from analytical consideration. The fiber tracheids and libriform fibers make up the bulk of the fibrous material in hardwood sheets. They are longitudinal components that differ in pitting from each other. The parenchymatous elements in hardwoods are arranged differently than softwoods but again their actual structure is not important to species analysis.

Species Identification

Microscopic techniques for species identification of softwood fibers are based almost entirely on longitudinal tracheids. Tracheids are the primary fibrous matter of softwood sheets. The pitting on these tracheids differs between species, allowing differentiation. Hardwood species are differentiated mainly by the structure of the vessel elements. Vessel elements from different species differ in both pitting and in shape.

Panshin and DeZeeuw's (1964) key for species analysis of woody fibers is designed for use with whole fibers. Several separations within the key are based either wholly or in part on fiber dimensions, especially fiber length. The refining operation in papermaking changes fiber length through transverse cutting, making accurate usage of those discriminations difficult. To avoid the error inherent in these discriminations the analyst chose one of two means, based on his judgement. The first was to look beyond the immediate separation and make the decision on the subsequent information. The second was to leave the analysis at that point. This was used in several cases that provided logical splits, such as the Southern hard pines. There are several separations in the hardwood section that are based on features not discernible in the samples. These were treated the same as the fiber dimension differentiations.

The use of the woody species key requires the knowledge of several chapters of Panshin and DeZeeuw's (1964) book. That information is condensed in the following outline and definitions:

Woody Fiber Species Analysis

I. Softwoods (coniferous)--three possible pit arrangements

- A. Pits resulting from tracheid and ray tracheid contact.
 - 1. Bordered
 - 2. Smaller than those from longitudinal tracheid contact.
 - 3. Generally at margin of ray crossing
 - 4. Attain best development in hard pines.
- B. Pits resulting from tracheid and tracheid contact.
 - 1. Bordered
 - 2. Large
 - 3. Numerous on radial walls
 - 4. Probably present in last few rows of the tangential walls of latewood tracheids.
- C. Pits resulting from tracheid and ray parenchyma contact (ray crossings). See Figure 2.
 - 1. Fenestriform (windowlike)
 - a. Large with broad apertures
 - b. Present in soft pines, red pine, scotch pine
 - 2. Pinoid
 - a. Smaller and more variable in size than fenestriform
 - b. More numerous per ray crossing
 - c. Present in all native hard pines other than red pine.
 - 3. Piceoid
 - a. Small bordered pits
 - b. Generally elliptical with a narrow, linear sometimes extended aperture.
 - c. Present in spruce, larch, Douglas fir.
 - 4. Taxodoid
 - a. Oval to circular apertures
 - b. Apertures much wider than narrow fairly even border.
 - c. Present in fir, Western Red Cedar, redwood, baldcypress.

Fenestriform



Pinoid
(can also be bordered)



Piceoid



Taxodioid



Cupressoid



Figure 2.--Structure of Softwood Pit Types.

5. Cupressoid
 - a. Resemble Piceoid
 - b. Aperture is included and elliptical, rather than linear.
 - c. Border remains wide
 - d. Present in cedars, yews, occasionally Hemlocks.

II. Hardwoods

- A. Vessel elements--the tangential diameter varies less than the radial.
 1. Perforation plates
 - a. Simple, one aperture
 - b. Scalariform
 1. ladderlike
 2. many holes, separated by bars of varying thickness.
 2. Pits
 - a. Contact between vessel element and ray parenchyma
 1. evident because size, nature, and arrangement different from other pits.
 2. bordered or simple.
 - b. Contact between vessel element and fiber or tracheid.
 1. generally in vertical rows
 2. bordered
 - c. Contact between vessel element and vessel element.
 1. conspicuous on tangential walls
 2. alternate
 - a. diagonal rows
 - b. circular to oval if uncrowded, polygonal or hexagonal if crowded.
 3. opposite
 - a. in horizontal rows or short horizontal rows
 - b. pits frequently rectangular
 4. scalariform
 - a. pits linear across the long axis of the vessel
 - b. pits arranged in ladderlike series
 3. Spiral thickening--ridges on the inner surface of the secondary wall.
- B. Tracheids
 1. Vascular
 - a. Similar in size and shape to small late-wood vessel elements
 - b. Closed at the ends
 - c. Walls contain numerous bordered pits

2. Vasicentric
 - a. Short, irregularly shaped cells
 - b. Tapering and rounded closed ends
 - c. Bordered pits on lateral walls

C. Fibers

1. Fiber tracheids
 - a. Thick-walled fibers with pointed ends
 - b. Bordered pits
2. Libriform fibers
 - a. Thick-walled fibers with pointed ends
 - b. Simple pits.

III. Definitions

- A. Confluent--flowing or running together so as to make one
- B. Contiguous--in contact
- C. Dentate--having toothlike projections
- D. Lenticular--shaped like a double-convex lens
- E. Ligulate extensions--tails at one or both ends of Hardwood vessel elements
- F. Mucronate--ending in a sharp point
- G. Procumbent--short wide ray parenchyma cells;
Upright--tall somewhat narrow ray parenchyma cells.
- H. Reticulum--a network
- I. Septate--a fiber with cross walls
- J. Uniseriate--rays of one cell in width.
bi-, multi-

The preceding outline is a reminder for the analyst, assuming he already has some basic knowledge. The analyst also needs standards for comparison. To offset the lack of extensive fiber libraries Carpenter and Leney's (1952) book was used. Presently, the woody fiber species analysis is only good on North American species.

The species analysis for non-woody fibers was carried out using TAPPI standard T-10 SM-41. All the information necessary for that analysis is in the standard. Corroborating evidence can be obtained using Isenberg's (1958) listing of stains and techniques.

LIST OF REFERENCES

- Buchanan, J. S. "Testing Methods for the Components of Fiberboard Cases," TAPPI, Volume 51 (February, 1968), p. 65-72.
- Carpenter, Charles H. and Leney, Lawrence. Paper Making Fibers. Meriden, Conn.: Meriden Gravure Co., 1952.
- Isenberg, Irving H. Pulp and Paper Microscopy. Appleton, Wisconsin: The Institute of Paper Chemistry, 1958.
- Johson, G. and Toroi, M. "Edge Crush Test for Corrugated Board" Paperboard Packaging, Vol. 7 (July, 1969), p. 35-38.
- McKee, R. C.; Gander, J. W.; and Wachuta, J. R. "Edgewise Compression Strength of Corrugated Fiberboard." Paperboard Packaging, Vol. 46 (November, 1961), p. 70-76.
- McKee, R. C.; Gander, J. W.; and Wachuta, J. R. "Flexural Stiffness of Corrugated Board." Paperboard Packaging (December, 1962), p. 111-118.
- Panshin, A. J. and DeZeeuw, C. Textbook of Wood Technology. New York: McGraw Hill Book Co., 1964.
- Ranger, A. E. "Evaluation of Fibrous Material for Board-making and Converting." Paper Technology, Volume 6 (1967), p. 246-249.
- Setterholm, V. C. and Gertjejansen, R. O. "Method for Measuring Edgewise Compressive Properties of Paper." TAPPI, Volume 48 (May, 1965), p. 308-313.
- Swedish Packaging Research Institute. "Edgewise Crush Resistance of Corrugated Fiberboard." Sweden, November, 1970. (Mimeographed.)

TAPPI Standard T10 SM-41. "Species Identification of Non-Woody Vegetable Fibers." Technical Association of the Pulp and Paper Industry (October, 1941).

TAPPI Standard T205 OS-71. "Forming Handsheets for Physical Tests of Pulp." Technical Association of the Pulp and Paper Industry (1971).

TAPPI Standard T233 SU-64. "Fiber Length of Pulp by Classification." Technical Association of Pulp and Paper Industry (1964).

TAPPI Standard T412 OS-63. "Moisture in Paper and Paper-board." Technical Association of Pulp and Paper Industry (1963).

TAPPI Standard T811. "Edgewise Compressive Strength of Corrugated Fiberboard (Short Column Test)." Technical Association of Pulp and Paper Industry (July, 1970).

MICHIGAN STATE UNIV. LIBRARIES



31293008709564