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The Effect of Variables on the Penetration of Starch Applied at the Size Press and the Relationship of Penetration to Strength Properties

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THE EFFECT OF VARIABLES ON THE PENETRATION
OF STARCH APPLIED AT THE SIZE PRESS AND THE
RELATIONSHIP OF PENETRATION TO STRENGTH PROPERTIES

A Thesis Submitted by

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In partial fulfillment for the requirements of a
Bachelor of Science Degree from
Western Michigan University
Kalamazoo, Michigan

To The Department of
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Western Michigan University

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ABSTRACT

A sheet internally sized with rosin was impregnated at the size press with starch under varying conditions of temperature, moisture, and speed.

Penetration was studied with a view to final sheet properties. A sliding microtome was used to cross-section the sheets. The sections thus obtained were extracted with hot water and hydrochloric acid. A photolorometric determination of starch was used utilizing the characteristic starch iodine reaction.

Increased temperature increased the pickup and penetration of starch. Increased moisture of the base paper initially aided pickup and penetration. High moistures tended to retard pickup and penetration. Increasing machine speed increased pickup and penetration. Starch on the surface seemed to improve burst more than an equal amount of starch within the base sheet. The addition of starch greatly reduced tear. The greater the penetration the greater the tear reduction.

LITERATURE REVIEW

INTRODUCTION

The literature was searched from 1964 to 1924. The search was primarily aimed at any studies done on penetration. Very few articles were found in this area. There was, however, considerable material concerning general background data about surface sizing, size presses, and size press variables.

DEFINITIONS OF SURFACE SIZING

Size can be defined as chemical other than bleach, fillers, pigments, and dyes which is added to the papermaking furnish, or subsequently applied after the web is formed, which alter the characteristics of a sheet that relate to its resistance to the passage of liquids which come into contact with the web. Sizing (1), then is the chemistry and processing of paper products to alter their resistance to the passage of liquids into and through them. Dreshfield's definition (above) points to two types of sizing, internal or beater sizing, and surface sizing.

Kumler mentions that sizing has been not too inaccurately called anything added to paper. Pattison (2) says that the process is called surface sizing when the web of paper is passed straight through the size press, sizing solution being applied to both sides of the web at the nip, where it is absorbed by the web.

Surface sizing is the application of sizing agents to the sheet of paper after it is formed (3). Killinger (4) notes that surface sizing of paper and board is a treatment applied to the paper surface to produce varying desired surface characteristics.

REASONS FOR SURFACE SIZING

Surface sizing can be used to improve finish, produce a surface better suited to printing, minimize scuffing, controlling sheet pore size, prevent excessive or undesirable penetration of other finishing agents, decorate or improve appearance, and improve strength characteristics (4).

Killinger (5) stresses increased strength as the primary purpose of size press treatment and considers increased resistance to the flow of liquids or gases as secondary. Witworth (6) on the other hand mentions water proofing, ink resistance, surface finish, erasability, and strength.

If a pigmented size is being used, an increase in brightness and/or opacity might also be achieved (5).

SIZE PRESS VARIABLES

There are four methods for machine application of surface size. There are the vertical size press, the horizontal size press, application at the calender stacks, and tub sizing.

In the vertical size press the sheet is passed horizontally through a vertical nip. Size is sprayed into the nip on the top surface, where it is pressed into the sheet. Size is applied to the lower surface by pickup of the bottom roll. Size is either sprayed on and/or picked up from a constant level vat, and carried to the nip where it is pressed into the sheet (6).

In a horizontal press the rolls are placed horizontal to one another. The size is normally applied with one spout on each side of the sheet with a small amount of excess running off each end (7).

Surface size can be applied at the calender on heavier weight papers. Generally a higher temperature and a higher viscosity size are applied at the calender (7).

Tub sizing consists of a tub or vat in which the web is immersed in size. The sheet is withdrawn and sent through a set of press rolls (3).

The vertical size press in recent years has acquired primary importance in this area (6). Therefore, the remainder of the discussion will be directed to this type of sizing.

One of the major variables of the size press itself is roll loading. With other factors constant, the higher the

nip pressure, the less amount of size pickup as the roll has a great effect on pickup. The greater the diameter the more nip pressure needed to squeeze out the same amount of size (7).

Roll hardness and material is probably the part of the design which has more differences of opinion than any other factor. About the only point of agreement is that the top and bottom rolls should be of different hardness. The top (hard) roll is often made of metal or hard rubber. The bottom roll is generally made of softer rubber. The softer the bottom roll the more size pickup (6).

Size press roll helper drives for the top roll prevent roll slippage and thus help reduce uneven applications due to roll slippage (8). Pickup may be increased by altering the speeds of the rolls relative to each other and the speed of the machine (6). The use of expander (Mount Hope) rolls to and from the press are important to prevent wrinkles (8). The use of teflon covered dryer rolls to prevent sticking on dryer cylinders widens the range of materials easily usable on the press (6). Dryer temperature and machine speed are important as they affect respectively sheet moisture and dwell time.

SIZE VARIABLES

As far as the size itself goes, the major variables are: choice of sizing agent(s); viscosity; temperature; ph; and

solids (9). Of these the major variables the important one as effecting penetration seems to be viscosity aside from the choice of sizing material itself (10). As the topic is stated, we are concerned with starch as a sizing material.

In North America, the trend has been to the use of corn starch conversions (18). In Europe the trend is to products produced from potatoe starch for sizing (18).

According to Strasser (19), chlorinated starches are best for all around surface sizing. This is because there is a lesser tendency of jelling than with other types of conversion, little or no stratification, and extremely good film, and little or no tendency to foam.

Oxidized starches, says Casey (20), produce results that cannot always be obtained from enzyme converted starches even though enzyme starch preparation has been perfected to the extent that excellent tub sizes can be prepared from enzyme conversion. Enzyme conversion is generally less expensive.

SHEET AND SIZE VARIABLES

Sheet density increases reduce the ability of the sheet to absorb. The greater the density the lower the pickup and the penetration (6). The amount of internal sizing is important. A hard sized (internally) sheet naturally will have lower pickup and penetration (6). Moisture is important. It is generally accepted that between 5 to 12 percent moisture is

optimum with an increase in moisture (within limits) causing an increase in penetration and pickup (11, 7, 6).

Cobb (13, 14) advanced the following equation for penetration:

$$L^2 = \frac{R S \cos. \theta t}{2 u}$$

L = depth of penetration, cm.
R = pore radius, cm.
S = surface tension liquid, dyne/cm²
 θ = contact angle, degrees
t = time of penetration, sec.
u = coefficient of viscosity, poises

Temperature should be kept as high as possible around 170-190°F (5). This would lower viscosity to increase penetration. Penetration is affected by the relationship of pore size and particle size of the starch (15). Some particles are too large to enter the sheet pores. They therefore stay on the surface of the web (15).

The sheet surface is made up of hills and valleys. This makes it particularly difficult to measure penetration distance (15).

Laboratory work done by Casey and Libby (12) indicated:

1. Depth of penetration of starch is decreased by increased sheet density, increased sizing, and decreased sheet moisture.
2. There appears to be a direct relationship between penetration and ink receptivity.
3. There is little relationship between penetration and wax number.

Dappen (15) did a study of starch clay coatings. His work indicated that more water (20 to 30 percent) was penetrated and less starch (3 to 4 percent) was penetrated into

the paper from the coating. He also noted that rapid drying could cause an increased rate of migration of the starch to the drying surface. He presented his photocolormetric method for determining a quantitative measure of starch present.

H. N. Lee (17) did a study on the characteristics of surface applied starch. He pointed out that penetration is greatly affected by internal sizing and quantity of size applied. "It appears to limit itself to the surfaces of the fibers and to filling more or less the interstices between the fibers." The starch did not appear to form a uniform unbroken film.

= Some very recent work (21) has been done in size press work by the Forest Products Laboratory. Starch was applied under varying conditions of starch type, viscosity, temperature, machine speed, and sheet moisture. The sheet was cut in two sections and stained with iodine solution. A visual observation of the stain was made to estimate degree of penetration. It was found that increased pickup generally improved physical tests. Starch which remained on the surface was more effective for burst and tensile than starch well penetrated. Major variables affecting penetration were viscosity, temperature, and machine speed. Increased viscosity tended to keep the starch from penetrating. Increased temperature and increased machine speed aided penetration.

LABORATORY OUTLINE

The purpose of this outline was to examine several variables in starch sizing; to study their effect on pickup and on penetration; and to relate any correlation between penetration and physical strength. To do this, the laboratory study falls into four areas:

1. Application of the sizing material.
2. Physical testing of the samples.
3. Analysis of starch distribution in samples.
4. Evaluation and correlation of data.

I. APPLICATION OF THE SIZING MATERIAL

This portion of the laboratory work was done in conjunction with the thesis of John Hartman (22) in the Spring of 1965. First, rolls of paper were ordered from the KVP Sutherland Paper Company to be made from the same run and to be internally sized with rosin. Sheet variables studied were different moisture levels.

The size used was a hydroxyethyl of starch, Penford gum 280. It was cooked in a continuous cooker built by Penick and Ford, Limited. The starch was varied in solids, temperature, and viscosity for different applications.

The size press on the Louis Calder Paper Machine at Western Michigan University was used for the application of

the starch. The machine variables studied were machine speed, drying temperature, and nip pressure.

Raw stock was KVP-Sutherland, 45 pounds (25 x 38 - 500), 85 percent jack pine, 15 percent broke, 1 percent rosin size.

II. PHYSICAL TESTING OF SAMPLES

The physical tests on the samples were tested by Wally Dean, and Hugh Meyers. All samples were tested for mullen, tensile, tear, fold, and Cobb size.

III. ANALYSIS OF STARCH DISTRIBUTION

To do this, Hartman's procedure was used with several variations. This consisted of a colorimetric determination of total starch in the sample to check pickup data; microtoming the sample; and running of a colorimetric determination of starch in the microtomed sections. The colorimetric determination of starch is essentially TAPPI Standard T 419 m60, and is as follows:

Apparatus

1. Disintegrator test tube and glass beads.
2. 50-ml. fritted glass filters.
3. Centrifuge with 50-ml capacity.
4. Spectrophotometer (Beckman DU).
5. Suction flask.
6. Three-way stop cock.
7. Hot plate.
8. 50, 100, 250, 500 ml. volumetric flasks.
9. 25 ml. graduate and 2.5 and 5 ml pipets.

Reagents

1. Hydrochloric acid: concentrated, 1:1 dilution and 1:10 dilution.
2. Potassium Iodide-Iodine reagent, ie: 7.5 g KI and 5 g I₂ per liter.
3. Cotton linters.

Note: For use in the microtomed samples the solution (2) will be diluted 1:50.

Test Specimen

A representative sample of paper weighing approximately one gram will be used for the total starch determination and 20 to 40 milligrams used for the microtomed section starch determination.

Procedure for Starch Determination

1. Transfer the specimen to the disintegrator and disintegrate in 30 ml. of distilled water.
2. Transfer to a 250 ml. beaker, using enough rinsing water to make the specimen up to 100 ml and heat on a hot plate to just below the boiling point for 15 minutes.
3. Transfer the contents to the suction crucible on the suction flask. Drain and wash with 5 ml. of hot water.
4. Turn the stop cock to cut off the suction. Blow air into the suction line to create a slight back pressure and turn the cock to seal it.
5. Add 12½ of the 1:1 HCl to the filtering crucible, allow to stand for 175-180 seconds and apply the suction.
6. Repeat steps four and five.
7. Reestablish back pressure and add 12½ ml of conc. HCl and allow to stand 15 to 20 seconds.
8. Wash the residue with 100 ml of hot water and test for complete removal of starch by adding a drop of dilute iodine solution.
9. Transfer the filtrate to a 250 ml volumetric flask, cool to room temperature, and dilute to the mark with water.

10. Pipet 25 ml. of the clear solution into a 50 ml volumetric flask, pipet 2.5 ml of the KI-I₂ sol. into the flask, dilute to the mark with water and mix thoroughly. Measure the absorbance at a wave length established by experiment on a Beckman DU, against a reference sample prepared identical to the test specimen except it will contain no starch. Read the starch concentration from a calibration curve.

Calibration Curve:

1. Weigh .1 (for total starch pickup) or .01 (for microtome samples) of starch corrected for moisture and ash.
2. Transfer to a 250 ml beaker, add 100 ml. distilled water and heat for 15 minutes just below boiling.
3. Add .02 g. cotton lintors to the solution and heat for 15 minutes more.
4. Do steps 3-10 for starch determination.
5. Remove aliquots to prepare the calibration curve.
6. Proceed with the preparation for absorbance measurements in step 11 for starch determination.

Sample cutting procedure: This was also essentially the same as that used by Hartman using a Spencer Mott sliding microtome and follows:

1. The microtome blade is mounted on the microtome.
2. A pine block with a surface dimension of $\frac{1}{2}$ x $\frac{1}{4}$ inches is mounted on the microtome sample holder. Another block is mounted just behind the first block on the sample holder so that the sample block will be under uniform mounting pressure.
3. The sliding microtome blade is then pulled across the block removing 5 micron sections until the surface of the block is smooth and parallel to the blade stroke. A final cut of two microns is then made to further smooth and level the block.
4. Samples of paper that are slightly smaller than the surface of the block are cut with a razor blade from the sheet to be tested.

5. A small quantity of Duco Cement is placed on the surface of the block.
6. Another pine block is then used to spread the cement over the surface of the block.
7. The cut paper sample is placed on the glued surface of the block.
8. A smooth glass plate is placed on the sample and a 200 gram weight is placed on the plate. The plate and weight are left on the paper for 70 seconds. This is to keep the sample smooth and level and give the glue time to bond the paper to the block.
9. The weight and glass are removed and an asbestos template is placed over the wood block. This is to shield the microtome parts from heat and reduce expansion. An infrared lamp is placed about $\frac{1}{2}$ inch over the sample and turned on for 70 seconds. This was found to be long enough to dry the glue, by Hartman.
10. The template and lamp are removed and the samples are then sectioned into 4 sections 10 microns thick. The various sections are collected in pre tared crucibles with a camel's hair brush.
11. The wood block is then leveled for the next sample.

TABLE I
CALIBRATION CURVE DATA

| <u>milliliters starch solution used</u> | <u>starch concentration milligrams per liter</u> | <u>absorbance</u> |
|---|--|-------------------|
| 2.0 | .9136 | .026 |
| 5.0 | 2.284 | .035 |
| 10.0 | 4.568 | .058 |
| 15.0 | 6.852 | .081 |
| 20.0 | 9.136 | .100 |
| 25.0 | 11.420 | .1175 |

11-mg./ liter

10-

9-

8-

7-

6-

5-

4-

3-

2-

1-

.01

.03

.05

.07

.09

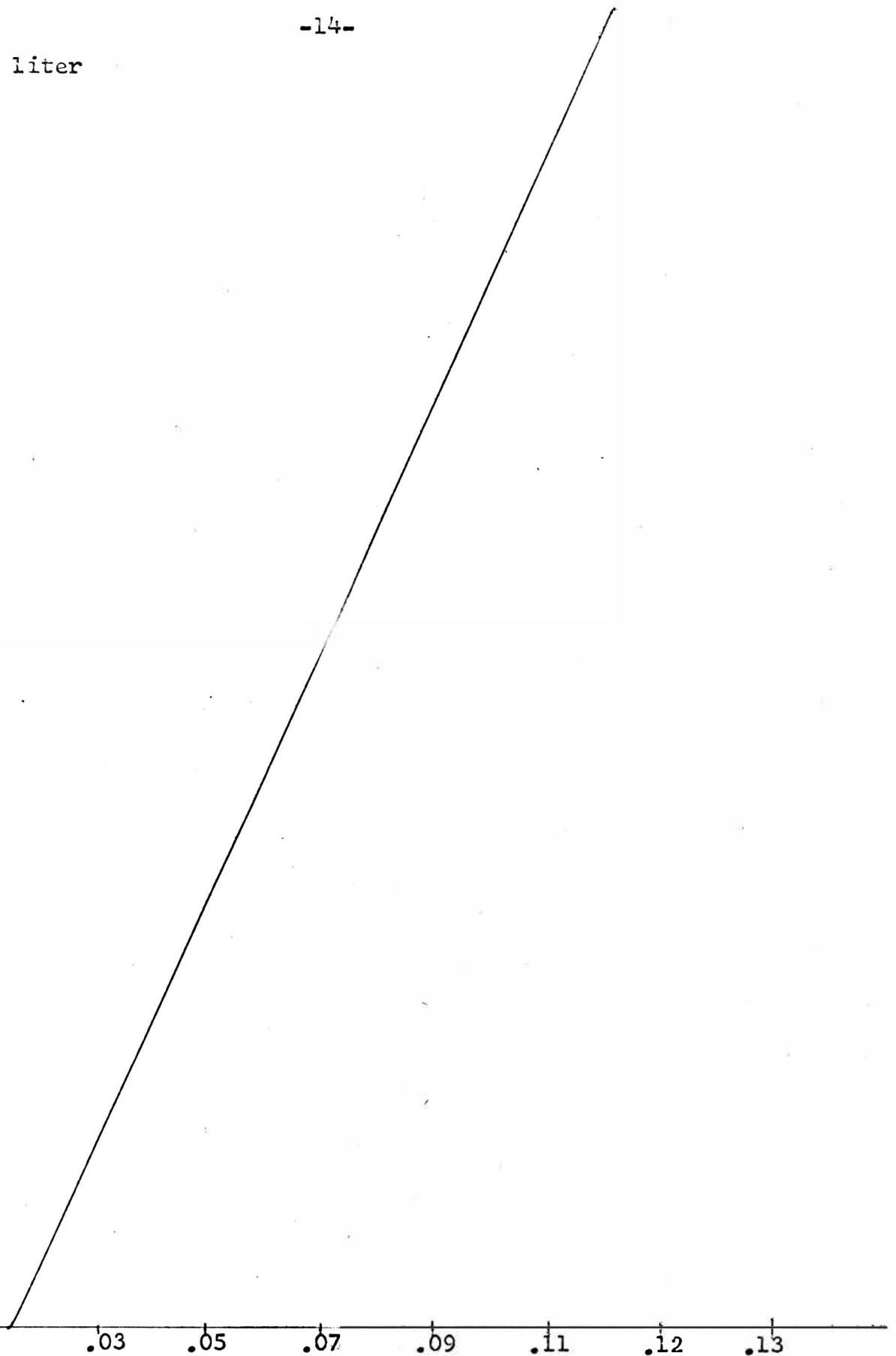
.11

.12

.13

Figure 1

CALIBRATION CURVE OF STARCH CONCENTRATION VS ABSORBANCE



DISCUSSION OF RESULTS

PENETRATION

TEMPERATURE VS PENETRATION (Figures 1, 2, 3)

Higher temperature seemed to give better relative penetration. At 130°F the starch concentration was high on the surface and went down going towards the center. At 140°F starch concentration was higher on the surface but the inside layers were much more even in starch concentration. At 147°F the first three layers were quite even in concentration with the 40 micron layer having a lower concentration of the starch.

MOISTURE VS PENETRATION (Figures 2, 5, 4)

As moisture (entering the press) went up, penetration went down with the starch seeming to pile up on the surface at 6.68 percent moisture (Figure 4). At 2.89 percent moisture there was more starch on the surface (Figure 2) with the inner layers fairly even in starch concentration. At 3.04 percent moisture the first two layers had higher concentrations and the inner two layers had lower concentrations of starch.

MACHINE SPEED VS PENETRATION (Figures 6, 2, 7)

Faster machine speed seemed to increase penetration. This could be due to the greater effective nip pressure created by the vector addition of vertical (nip) and horizontal (speed) forces.

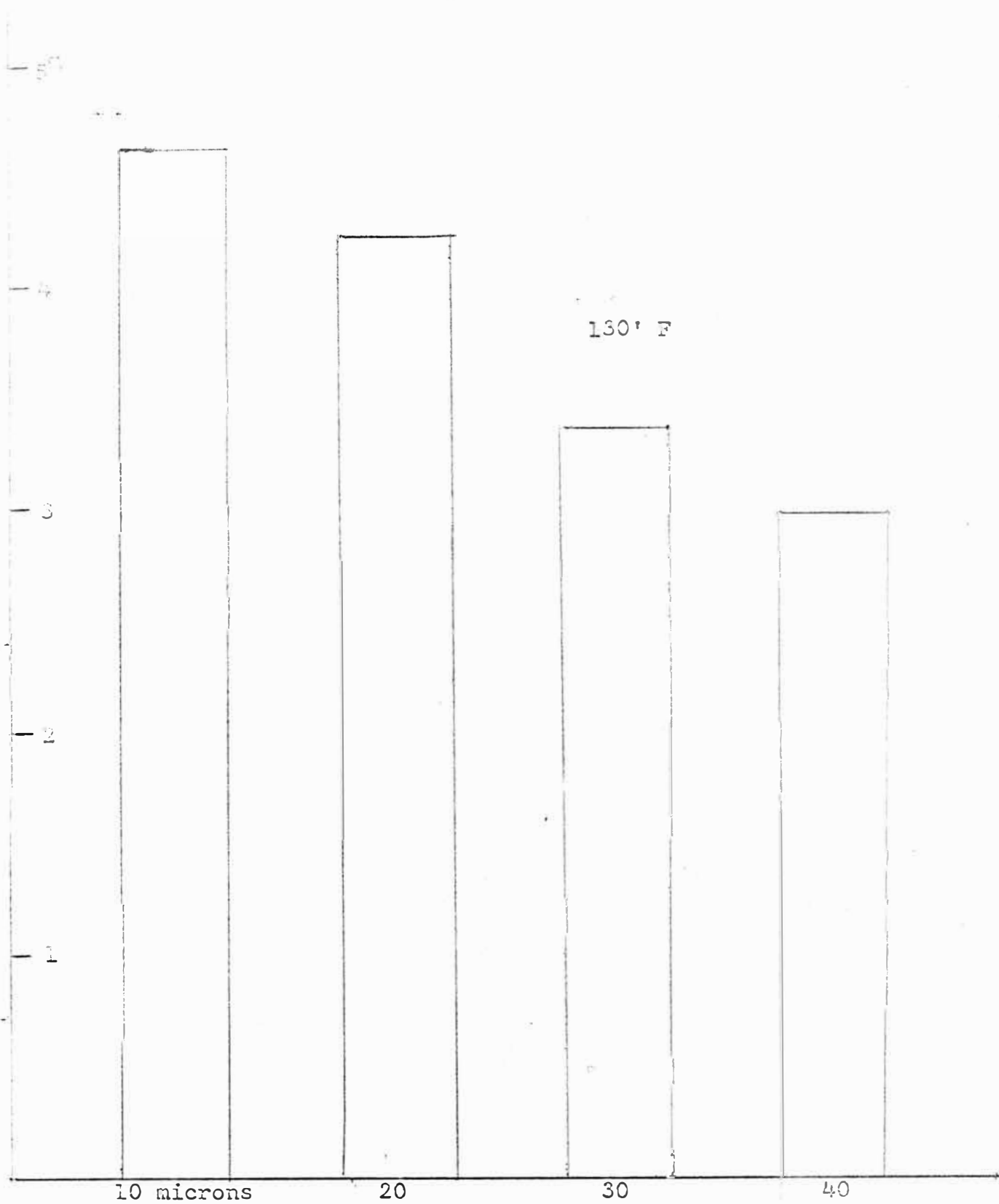


Figure 2

PERCENT STARCH VS PENETRATION, RUN 1

140°F.
250 FPM
2.89% moisture

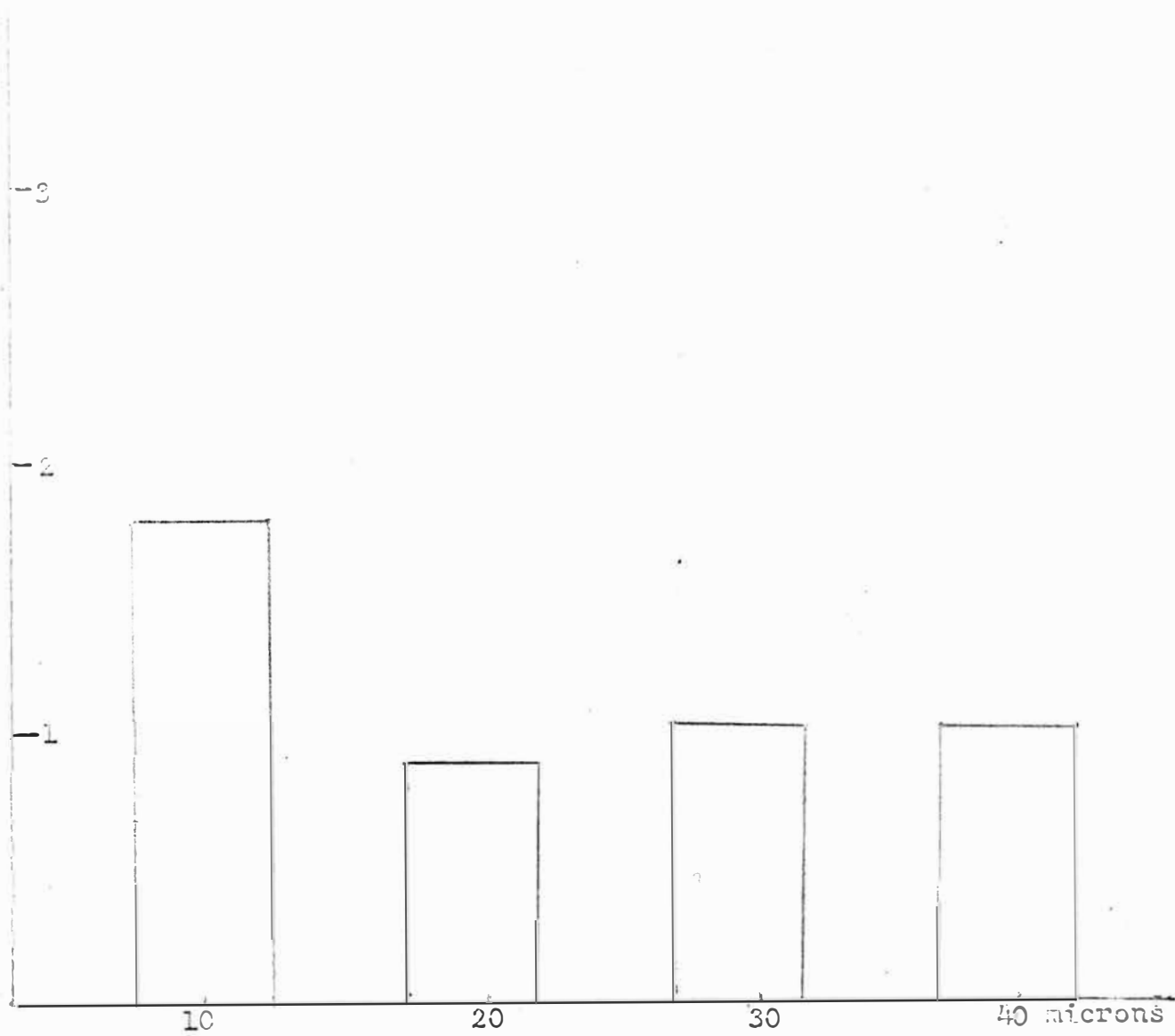


Figure 3

PERCENT STARCH VS PENETRATION, RUN 2

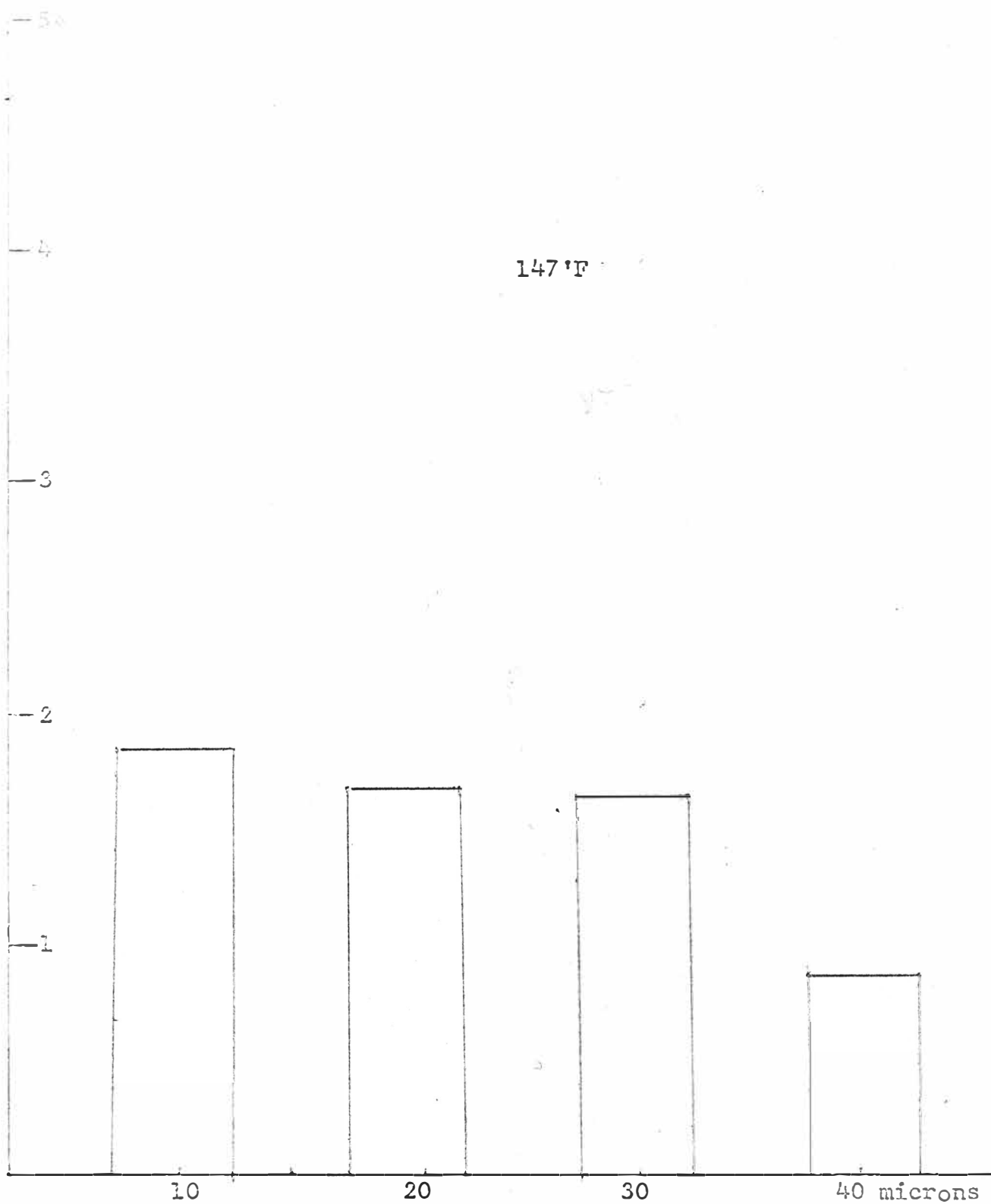


Figure 4

PERCENT STARCH VS PENETRATION, RUN 3

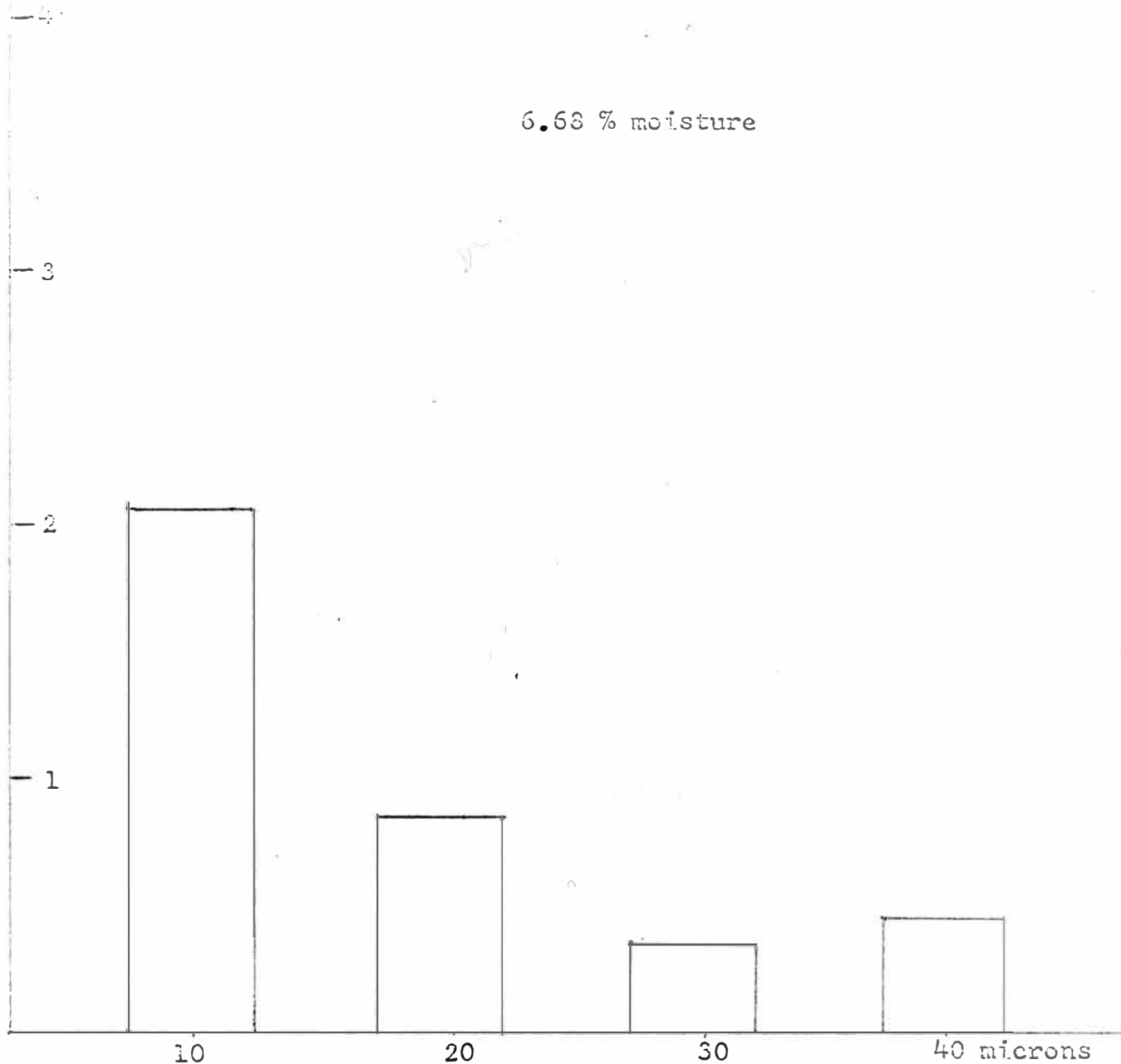


Figure 5

PERCENT STARCH VS PENETRATION, RUN 4

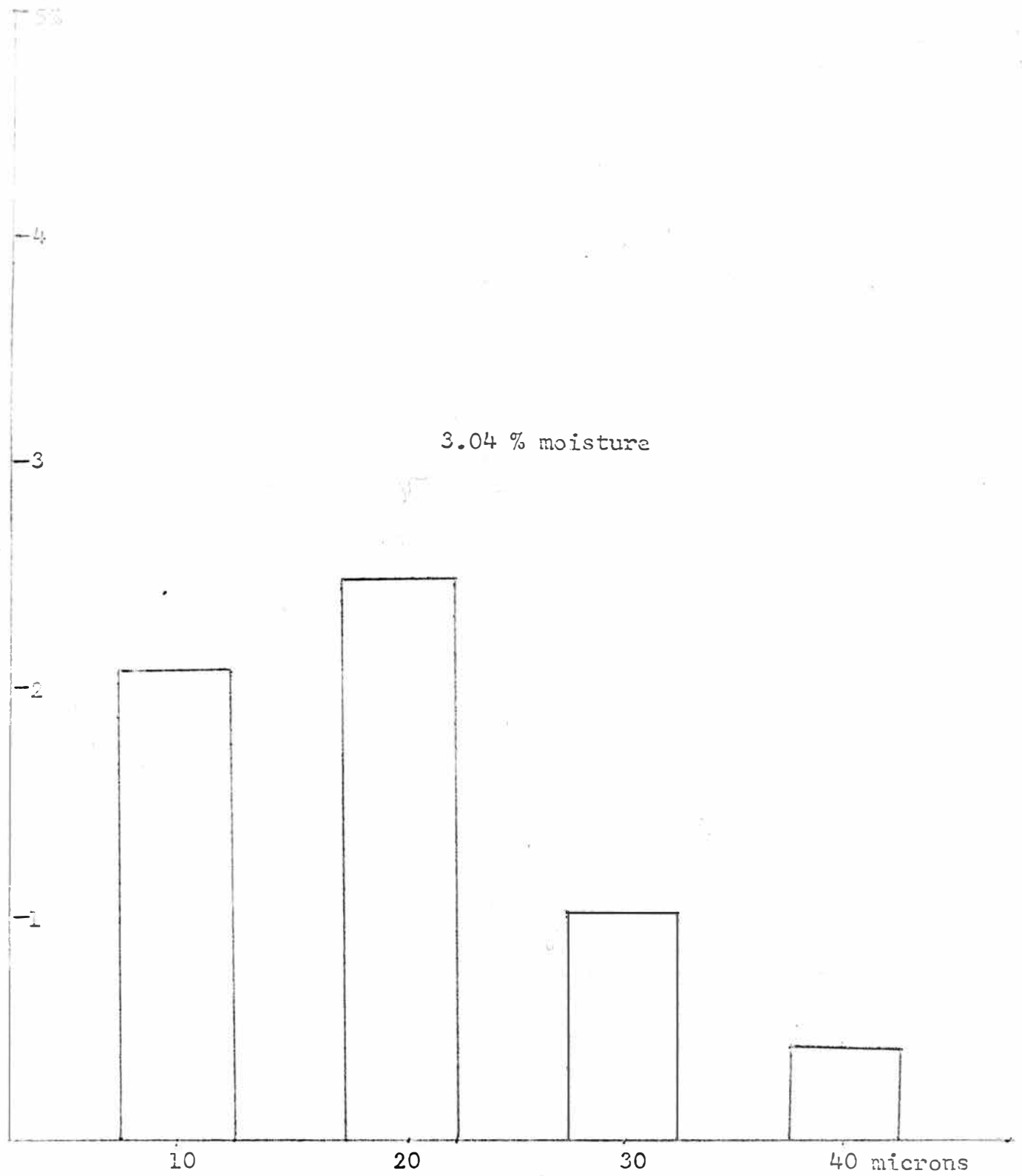


Figure 6

PERCENT STARCH VS PENETRATION, RUN 5

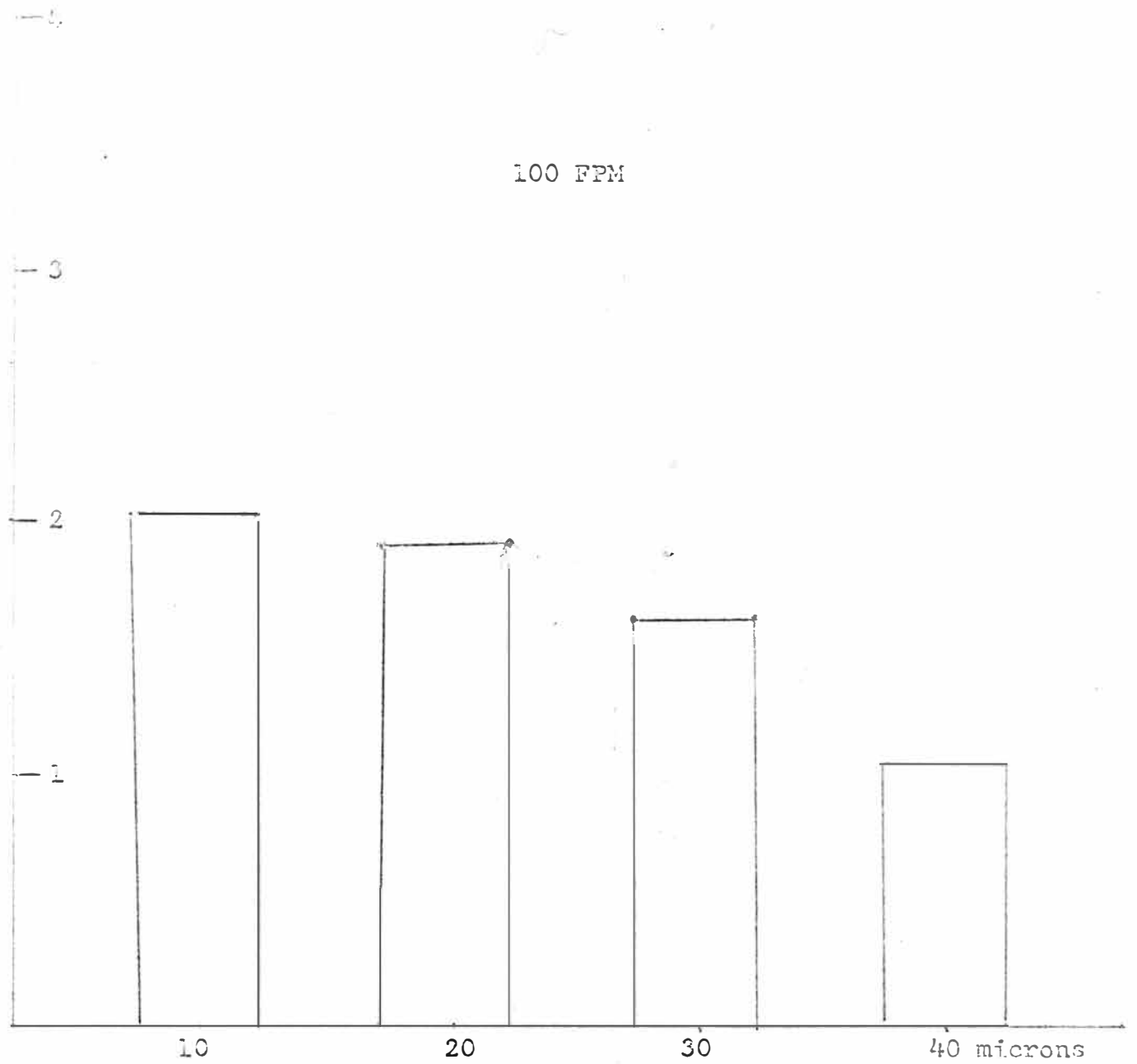


Figure 7

PERCENT STARCH VS PENETRATION, RUN 6



Figure 8

PERCENT STARCH VS PENETRATION, RUN 7

TABLE II
MACHINE RUN DATA

| | Starch Solids | Starch Temp. | Dudley Viscosity | Moisture Content | Sheet Temp. | Machine Speed | Nip Loading | Dryer Temp. | Consumption lb/ream |
|----|------------------|-----------------|---------------------|---------------------|----------------|------------------|----------------|----------------|------------------------|
| 1 | 11.65% | 130°F | 83 sec | 2.89% | 195°F | 250fpm | 18psi | 210°-210°F | .689 |
| 2 | 11.65 | 140 | 71 | 2.89 | 195 | 250 | 18 | 210-210 | 2.42 |
| 3 | 11.65 | 147 | 64 | 2.89 | 190 | 250 | 18 | 210-210 | 4.74 |
| 4 | 11.65 | 140 | 67 | 6.68 | 190 | 250 | 18 | 215-215 | 1.84 |
| 5 | 11.65 | 140 | 67 | 3.04 | 200 | 250 | 18 | 215-215 | 3.16 |
| 6 | 11.65 | 140 | 67 | 2.89 | 150 | 100 | 18 | 215-215 | .615 |
| 7 | 11.65 | 140 | 67 | 2.89 | 185 | 400 | 18 | 215-215 | 4.01 |
| 8 | 11.65 | 140 | 67 | 2.89 | 200 | 250 | 8 | 220-220 | 2.55 |
| 9 | 11.65 | 140 | 67 | 2.89 | 190 | 250 | 25 | 215-220 | 2.38 |
| 10 | 15.05 | 150 | 151 | 2.78 | 190 | 250 | 18 | 220-220 | 3.16 |
| 11 | 15.05 | 140 | 236 | 2.46 | 205 | 250 | 18 | 220-220 | 3.59 |
| 12 | 15.05 | 130 | 360 | 2.42 | 200 | 250 | 18 | 220-220 | 3.73 |
| 13 | 8.88 | 150 | 51 | 2.43 | 200 | 250 | 18 | 220-220 | 1.57 |
| 14 | 8.88 | 140 | 59 | 2.43 | 195 | 250 | 18 | 220-220 | 1.61 |
| 15 | 8.88 | 130 | 64 | 2.43 | 200 | 250 | 18 | 220-220 | 1.65 |

Starch cook 1-3

starch cook: 232°F

steam flow: 27%

Starch cook 4-15

starch cook: 234°F

steam flow: 27%

PICKUP

Pickup was measured by take up of the starch in the machine room. An attempt was made to relate this to the total starch found in each of the sections for each run. No correlation could be drawn between the two. This could be due to microtoming. The sheet does not always microtome 10 microns at a time and the weights of the layers are not always equal. Or, it could be due to other factors in the procedure.

An attempt was made to run a TAPPI Standard (T 419 m-60) determination of total starch in each run using one gram samples. It was found (during the last week) that the photomultiplier in the Beckman DU spectrophotometer was unreliable and definitely defective during the period of these runs. Because the defect was not discovered until it was too late to do the work over, it was only possible to rerun one sample that had not been discarded. This was run number ten. The results of the spectrophotometric absorption were very close to the pickup data from the machine room (6.52 vs 6.63 percent).

Machine room pickup data will be used for total pickup in this paper.

TEMPERATURE VS PICKUP

An increase in temperature was found to increase percent pickup. At 130°F, 1.53 percent starch was picked up. At 140°F, 5.14 percent starch was picked up. At 147°F, 9.62 percent starch was picked up.

MOISTURE VS PICKUP

An increase in moisture of the sheet initially improved pickup. A further increase in sheet moisture retarded starch pickup.

| <u>Sheet Moisture</u> | <u>% Pickup</u> |
|-----------------------|-----------------|
| 2.9 % | 5.14 |
| 3.04% | 6.6 |
| 6.68% | 4 |

NIP PRESSURE VS PICKUP

Increasing nip pressure reduced pickup. A proportionate reduction in burst was also evident.

| <u>Burst (psi)</u> | <u>Nip Pressure (psi)</u> | <u>% Pickup</u> |
|--------------------|---------------------------|-----------------|
| 45.1 | 8 | 5.41 |
| 41.7 | 18 | 5.14 |
| 37.4 | 25 | 4.76 |

DISCUSSION OF PHYSICAL DATA

Physical data is particularly hard to correlate to pickup or to penetration because different pickup levels were accompanied by different degrees of penetration. As both variables (pickup and penetration) were uncontrolled, great difficulty is encountered in trying to study the effect of either variable.

The following conclusions were drawn. A little starch on the surface (2 percent) seemed to do more for burst improvement than relatively larger amounts well dispersed in the sheet.

TABLE III

WMU SIZE PRESS PROJECT 1965
Physical and Optical Test Results

Summary

| <u>Run No.</u> | <u>(1)</u> | <u>(2)</u> | <u>(3)</u> | <u>(4)</u> |
|---|------------|-------------------|------------|------------|
| O.D. Basis Wt. (g/m ²) (Moist. Free) | 72.4 | 72.4 | 72.3 | 73.1 |
| Tensile MD. | 19.0 | 20.5 | 22.4 | 22.0 |
| Tensile C.M. (#/15mm) | 11.4 | 11.8 | 11.7 | 10.1 |
| Breaking Len. (M.D., in M) | 7,940 | 8,570 | 9,540 | 9,110 |
| B.L. C.M. (in M) | 4,760 | 4,930 | 4,890 | 4,170 |
| Fold M.D. | 430 | 376 | 318 | 327 |
| Fold C.M. (MIT) | 444 | 302 | 297 | 302 |
| Tear M.D. (Tappi Corr.) | 85.4 | 87.5 | 87.9 | 88.6 |
| Tear C.M. | 95.7 | 99.1 | 96.9 | 103 |
| Mullen (Tappi Corr.) | 37.4 | 41.7 | 38.4 | 38.7 |
| Stiffness M.D. (Gurley) | 4.73 | 5.43 | 5.40 | 5.3 |
| Stiffness C.M. | 2.92 | 3.48 | 3.20 | 2.8 |
| Brightness W.S. (I.P.C.) | 80.0 | 80.2 | 79.7 | 80.0 |
| Brightness Felt Side | 81.7 | 81.8 | 81.0 | 81.5 |
| Opacity Wire Side (Tappi) | 88.0 | 88.0 | 87.0 | 89 |
| Opacity Felt Side | 88.0 | 88.0 | 87.0 | 90 |
| Gloss W.S. (B & L%) | 14 | 15 | 14 | 14 |
| Gloss Felt Side | 15 | 14 | 13 | 13 |
| Starch Pickup % | 1.53 | 5.14 (Hartman) | 9.62 | 3.92 |

TABLE III (Continued)

WMU SIZE PRESS PROJECT 1965
Physical and Optical Test Results

Summary (cont.)

| <u>Run No.</u> | <u>(5)</u> | <u>(6)</u> | <u>(7)</u> | <u>Control</u> |
|---|------------|------------|------------|----------------|
| O.D. Basis Wt. (g/m ²) (Moist. Free) | 74.0 | 73.5 | 74.1 | 66.6 |
| Tensile M.D. | 22.4 | 22.1 | 22.5 | 16.4 |
| Tensile C.M. (#/15mm) | 9.7 | 9.1 | 10.6 | 9.4 |
| Breaking Len. (M.D., in M) | 9,150 | 9,100 | 9,180 | 7,446 |
| B.L. C.M. (in M) | 3,970 | 4,080 | 4,330 | 4,264 |
| Fold M.D. | 447 | 312 | 416 | 112 |
| Fold C.M. (MIT) | 471 | 284 | 417 | 63 |
| Tear M.D. (Tappi Corr.) | 87 | 93 | 82 | 140 |
| Tear C.M. | 103 | 112 | 94 | 155 |
| Mullen (Tappi Corr.) | 37.4 | 40.1 | 42.3 | 25.9 |
| Stiffness M.D. (Gurley) | 5.5 | 5.8 | 4.8 | 4.4 |
| Stiffness C.M. | 2.8 | 3.1 | 2.6 | 2.7 |
| Brightness W.S. (I.P.C.) | 80.0 | 79.8 | 79.3 | 81.4 |
| Brightness Felt Side | 81.2 | 81.2 | 80.8 | 82.8 |
| Opacity W.S. (Tappi) | 88.0 | 88.0 | 87.0 | 89 |
| Opacity Felt Side | 88.0 | 88.0 | 87.0 | 90 |
| Gloss W.S. (B & L%) | 14 | 12 | 17 | 8 |
| Gloss Felt Side | 14 | 13 | 16 | 7 |
| Starch Pickup % | 6.61 | 1.36 | 8.25 | ---- |

With the very high (8 percent) starch pickup, a well dispersed starch was as effective as a low pickup in which the starch remained on the surface. This trend was much harder to distinguish in tensile. Here greater pickup, in most cases produced somewhat higher tensile values.

No conclusive correlation could be drawn with fold. Fold was greatly improved by the application of 1.53 percent starch. As pickup increased fold sometimes increased and sometimes decreased. This did not seem to be dependent upon degree of penetration.

Greater penetration and greater pickup tended to reduce tear. The mere application of starch created a significant reduction in tear. This relatively large reduction was not proportionately repeated by increasing amount of starch application.

Stiffness increased with increased pickup and starch on the surface was more effective in increasing stiffness. This should be expected because starch is stiffer than the fiber. If starch is concentrated in any area (in this case on the surface) it can cover whole fibers or sections of fibers, thereby preventing the bending of the fiber.

It may be noted that physical test results concerning degree of penetration agreed very well with those results obtained by Chilson and Fahey (21) at the Forest Products Laboratory.

DISCUSSION OF OPTICAL DATA

Brightness was slightly reduced by application of starch and further reduced by increasing pickup. Gloss and opacity showed no significant trends.

TABLE IV
STARCH ANALYSES OF MICROTOME SAMPLES

| <u>Run No.</u> | <u>Absorbance</u> | <u>Starch, mg.</u> | <u>O.D. Paper, mg.</u> | <u>Starch, %</u> |
|----------------|-------------------|--------------------|----------------------------|------------------|
| 1 F-1 | .047 | .68 | 15.1 | 4.64 |
| 2 | .030 | .32 | 7.61 | 4.22 |
| 3 | .031 | .34 | 11.8 | 3.38 |
| 4 | .040 | .536 | 15.6 | 3.01 |
| 1 W-1 | .034 | .4 | 8.37 | * |
| 2 | .031 | .34 | 8.0 | |
| 3 | .031 | .34 | 8.3 | |
| 4 | .022 | .14 | 6.76 | |
| 2 F-1 | .025 | .21 | 20.4 | 1.73 |
| 2 | .020 | .10 | 23.1 | .856 |
| 3 | .017 | .03 | 9.65 | 1.03 |
| 4 | .017 | .03 | 7.14 | 1.00 |
| 2 W-1 | .032 | .36 | 12.5 | |
| 2 | .026 | .226 | 15.0 | |
| 3 | .023 | .17 | 9.7 | |
| 4 | .021 | .12 | 7.85 | |
| 3 F-1 | .036 | .444 | 21.1 | 1.835 |
| 2 | .026 | .226 | 16.4 | 1.66 |
| 3 | .022 | .140 | 6.56 | 1.62 |
| 4 | .022 | .140 | 8.65 | .861 |
| 3 W-1 | .026 | .226 | 15.4 | |
| 2 | .024 | .186 | 8.37 | |
| 3 | .026 | .226 | 15.4 | |
| 4 | .017 | .03 | 11.1 | |
| 4 F-1 | .034 | .4 | 15.6 | 2.04 |
| 2 | .019 | .08 | 7.6 | .849 |
| 3 | .017 | .03 | 7.32 | .38 |
| 4 | .017 | .03 | 11.2 | .457 |
| 4 W-1 | .021 | .122 | 10 | |
| 2 | .018 | .056 | 16.2 | |
| 3 | .017 | .030 | 8.45 | |
| 4 | .018 | .056 | 7.6 | |

* Felt and wire side are averaged for percent starch.

TABLE IV (Continued)
STARCH ANALYSES OF MICROTOME SAMPLES

| <u>Run No.</u> | <u>Absorbance</u> | <u>Starch, mg.</u> | O.D. | <u>Starch, %</u> |
|----------------|-------------------|--------------------|-------------------|------------------|
| | | | <u>Paper, mg.</u> | |
| 5 F-1 | .020 | .10 | 7.52 | 2.06 |
| 2 | .021 | .122 | 4.94 | 2.47 |
| 3 | .022 | .14 | 8.85 | 1.00 |
| 4 | .017 | .030 | 7.71 | .394 |
| 5 W-1 | .034 | .4 | 16.8 | |
| 2 | .037 | .47 | 16.9 | |
| 3 | .017 | .030 | 8.18 | |
| 4 | .017 | .03 | 7.52 | |
| 6 F-1 | .022 | .140 | 9.68 | 2.03 |
| 2 | .021 | .122 | 6.72 | 1.90 |
| 3 | .017 | .03 | 4.59 | 1.60 |
| 4 | .017 | .03 | 4.16 | 1.04 |
| 6 W-1 | .024 | .186 | 6.37 | |
| 2 | .021 | .122 | 6.12 | |
| 3 | .021 | .122 | 4.92 | |
| 4 | .018 | .056 | 4.11 | |
| 7 F-1 | .055 | .856 | 19.3 | 3.87 |
| 2 | .045 | .680 | 11.3 | 5.02 |
| 3 | .036 | .444 | 7.62 | 4.25 |
| 4 | .032 | .360 | 6.09 | 3.88 |
| 7 W-1 | .040 | .534 | 16.6 | |
| 2 | .034 | .400 | 18.2 | |
| 3 | .027 | .256 | 9.8 | |
| 4 | .028 | .272 | 10.3 | |

SAMPLE CALCULATIONS OF RESULTS

I. Weight of starch in milligrams for calibration curve:

Weight of air dry starch (g) X % moisture = weight
of oven dry starch (g)

.0125 g air dry starch X 8.65% moisture =

.01142 g oven dry starch or 11.42 mg oven dry starch

II. Weight of starch per liter for calibration curve:

$$\frac{\text{mg O.D. starch}}{250 \text{ ml dilution}} \times \frac{\text{ml aliquot used}}{100 \text{ ml dilution}} \times \frac{1000 \text{ ml}}{\text{liter}} = \frac{\text{mg O.D. starch}}{\text{liter}}$$

(From 5 milliliter aliquot)

$$\frac{11.42 \text{ mg starch}}{250 \text{ ml}} \times \frac{5 \text{ ml starch sol.}}{100 \text{ ml}} \times \frac{1000 \text{ ml}}{\text{liter}} = \frac{\text{mg O.D. starch}}{2.284 \text{ liter}}$$

III. Weight of starch in microtome sample:

The absorbance reading of the microtome sample can
be used
be used to determine the concentration of starch in the
sample by taking the coordinate of the absorbance reading
from the calibration curve.

$$\frac{\text{mg starch}}{\text{liter}} \times \frac{200 \text{ ml sample}}{1000 \text{ ml}} \times \frac{1 \text{ liter}}{1000 \text{ ml}} = \frac{\text{mg starch}}{\text{in sample}}$$

(From sample 1 F-1)

The absorbance reading of .047 gives 3.4 mg/l starch as
the concentration of starch in the sample.

$$\frac{3.4 \text{ mg starch}}{\text{liter}} \times \frac{200 \text{ ml sample}}{1000 \text{ ml}} \times \frac{1 \text{ l}}{1000 \text{ ml}} = \frac{68 \text{ mg starch}}{\text{in sample}}$$

IV. Percent starch in paper:

1) Weight air dry paper X percent moisture =
weight oven dry paper.

2) From 1 F-1 and 1 W-1 sample.

$$\frac{\text{mg starch in 1 F-1} + \text{mg starch in 1 W-1}}{\text{mg paper in 1 F-1} + \text{mg paper in 1 W-1}} \times 100\% = \frac{\% \text{ starch}}{\text{in paper}}$$

$$\frac{.68 \text{ mg starch in 1 F-1} + .4 \text{ mg starch in 1 W-1}}{15.1 \text{ mg paper in 1 F-1} + 8.37 \text{ mg paper in 1 W-1}} \times 100\% = \frac{4.64\%}{\text{starch}}$$

CONCLUSIONS

Starch can be effectively used at the size press to improve physical properties of paper.

Altering variables such as viscosity, temperature, machine speed, and moisture, penetration may be promoted or retarded. Greatest improvement in tensile, burst, and stiffness result with the starch on the surface. The application of starch significantly reduces tear.

SUGGESTIONS FOR FURTHER WORK

A study of toughness using the integrator available with the Instron Tester should be a valuable look in the physical testing area. Further work could also be done on improving the method of sectioning samples or in developing a new method.

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