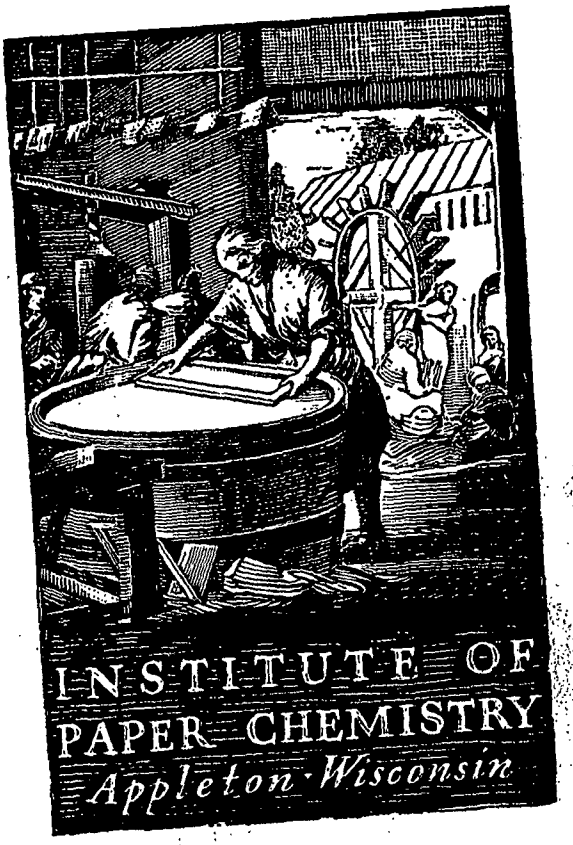

STUDIES ON THE FACTORS GOVERNING RETENTION AND EFFECTIVENESS OF STARCH XANTHATES AND XANTHIDES BY WOOD PULP IN PAPERMAKING

Project 2580

Report Seven

A Quarterly Report

to

AGRICULTURAL RESEARCH SERVICE
UNITED STATES DEPARTMENT OF AGRICULTURE

June 15, 1967

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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STUDIES ON THE FACTORS GOVERNING RETENTION AND EFFECTIVENESS OF STARCH XANTHATES AND XANTHIDES BY WOOD PULP IN PAPERMAKING

SUMMARY

Short-term aging effects on ex situ starch xanthide were examined on a small experimental paper machine. Adding starch xanthide directly from the continuous cross-linking unit to the wood fiber suspension produced a more freely draining stock than when the xanthide is stored 15 minutes before blending with the fiber. In respect to paper performance, aged xanthide was equal to or superior to the unaged material, except in regard to xanthide retention efficiency and wet tensile strength. In the case of 16-hour soaking wet breaking length, paper treated with aged xanthide was equal to the unaged trial paper.

Microscopic examination of paper prepared in an experiment described in the preceding report, shows that xanthide aggregates are present in paper prepared from ex situ starch xanthide stored 4 and 24 hours at 20°C. These aggregates appear to make little contribution to paper strength properties. Visual evidence is presented that starch xanthide which does improve the physical strength of paper is deposited as a layer on the surface of the fiber. There are indications that xanthide sorbed on the fiber is poorly detected by the usual iodine--potassium iodide stain. Graff's "C" stain, which contains aluminum, zinc, and calcium chlorides, in addition to iodine and potassium iodide, is more effective.

Experimental work for a study relating sorption temperature and alum concentration to starch xanthide retention rates has been completed but the data are only partially evaluated. Lower temperatures and higher alum concentrations appear to favor starch xanthide retention.

INTRODUCTION

The work being done as Institute Project 2580 is carried out in cooperation with the Agricultural Research Service of the United States Department of Agriculture. The scope is specified in Contract Number 12-14-100-8308(71) issued under the authority of the Research Marketing Act of 1946 as amended. The purpose of this project is to expand the use of cereal products in papermaking by determining the factors governing sorption of starch xanthate and starch xanthide by wood fibers in dilute aqueous suspensions. This report is the seventh issued quarterly under the terms of the contract.

The work covered in the preceding report represented a change in approach from dealing with starch xanthide prepared in the presence of the fiber (in situ) to starch xanthide prepared separately from the fiber (ex situ). This was done in order to investigate the factors affecting starch xanthide retention separately from those affecting the formation of starch xanthide by the oxidative coupling of starch xanthate. In addition, the shift of emphasis brings this study more closely in line with the research program of the sponsoring agency. The present report continues the investigation of ex situ starch xanthide.

EVALUATION OF SHORT-TERM AGING OF EX SITU STARCH XANTHIDE

A laboratory procedure has been developed for preparing starch xanthide separately from the fiber (ex situ) and has been evaluated (1). It was found that such xanthide dispersions have a useful storage life of from 5 to 60 minutes. That is, properties of handsheets are not different if the xanthide is used 5 or 60 minutes after it is formed.

During a conference last April, involving the research teams of the co-operating institutions, concern was expressed about possible differences between xanthide used immediately after cross-linking and that used after being stored 5 to 60 minutes. The work being done in the cooperator's laboratory is with unaged starch xanthide. Since it was not feasible to evaluate starch xanthide aging effects between 0 to 5 minutes in the laboratory, an alternative method was evolved.

Starch xanthide was prepared by a continuous process and added to the fiber suspension either directly from the cross-linking unit or added after aging for 15 minutes. The stock was blended with the xanthide 5 minutes and then diluted to operating consistency and formed into paper on a small experimental machine.

The procedures and proportions used for continuous cross-linking were based on those described in Reference (2). However, pH control during this study was achieved by acidifying both the xanthate and hypochlorite solutions to about pH 5. By doing this, control of only one variable, the hypochlorite flow, was necessary during the cross-linking process.

Starch xanthide was prepared continuously by bleeding a stream of acidified sodium hypochlorite into a stream of dilute starch xanthate. The flow of the hypochlorite was restricted to the level that produced the blue color of the starch--iodine complex in the combined streams.

The starch xanthate solution was prepared by diluting 1763 g. of xanthate Run No. 502 (181.6 g. xanthate starch, D.S. 0.13) with 90 liters of deionized water in a 30-gallon tank. A Lightnin' mixer provided agitation. To this solution (0.2 g./100 ml.) were added 73 ml. 10% potassium iodide [4% on the xanthate as in Reference (2)] and enough 20% acetic acid to lower the pH to 5.0 to 5.2.

To prepare the cross-linking reagent, commercial household bleach (Hi-lex) was diluted with deionized water to give 2 liters of 1% sodium hypochlorite. It was then acidified to pH 5.0-5.2 with 20% acetic acid just before use.

The cross-linking unit consisted of a Jabsco pump (Model No. 3010, phenolic body, 3/8-in. ports) with a 30-mm. by 190-mm. glass mixing chamber on the inlet side and a segment of 8-mm. glass tubing on the outlet side. Connections were made with 3/8-in. I.D. rubber tubing.

The mixing chamber was preceded by an 8-mm. glass "Y" tube where the two liquid flows joined. Tygon tubing was used to connect one arm of this tube directly to a throttling valve and then to an elevated 2-liter separatory funnel which contained the hypochlorite solution. Fastened to the other arm were several feet of 3/8-in. I.D. rubber tubing. The free end was immersed in the xanthate solution.

The glass tube on the outlet side of the pump was intended to provide an opportunity for visual control of the hypochlorite flow. In practice, this tube was not used for that purpose. The color change was observed in the mixing chamber ahead of the pump. The velocity drop produced by increasing the diameter of the line from about 8 to 30 mm., was sufficient to mix thoroughly the two streams so that the color change occurred in the first 20 to 30 mm. of the chamber. A little

less than 8 minutes were required to cross-link the entire 91.8-liter batch of 0.2% starch xanthate solution.

The appearance of the starch xanthide preparation was dominated by the blue color formed by the starch moiety and the iodine produced from potassium iodide by the small excess of hypochlorite. This color persisted through forming the wet web and was noticeable in remoistened fresh paper. The xanthide pH was very nearly the same as that of the xanthate and hypochlorite solutions. No sediment of coagulated xanthide was found after 15 minutes of storage.

The Institute of Paper Chemistry Continuous Web Former is a small experimental paper machine about one foot wide. It uses a flow spreader followed by a vacuum forming box to establish the web. A transfer felt applies the wet web onto a small steam-heated Yankee drier to produce a machine-finish product. A schematic flow diagram is shown in Fig. 1. Figures 2 and 3 are photographic views of the equipment. A standard 72 x 56 semitwill bronze wire was used for these experiments.

Four-pound (o.d.) portions of the same Rayonier bleached kraft pulp used throughout this project were soaked overnight in filtered tap water and beaten to 700 Schopper-Riegler freeness in a 5-lb. Valley beater. The refined stock was pumped over to the machine chest (see Fig. 1) and treated with 2 g. alum per 100 g. fiber. For the control, Run No. I, the stock was diluted to the 0.05% operating consistency without further treatment. In Run No. II, starch xanthide was pumped, as formed, directly into the machine chest (1.7% consistency) and blended with the fibers 5 minutes before the mixture was diluted to operating consistency. In Run No. III, the starch xanthide was pumped as it was formed into several stainless steel containers and allowed to stand for 15 minutes after production was

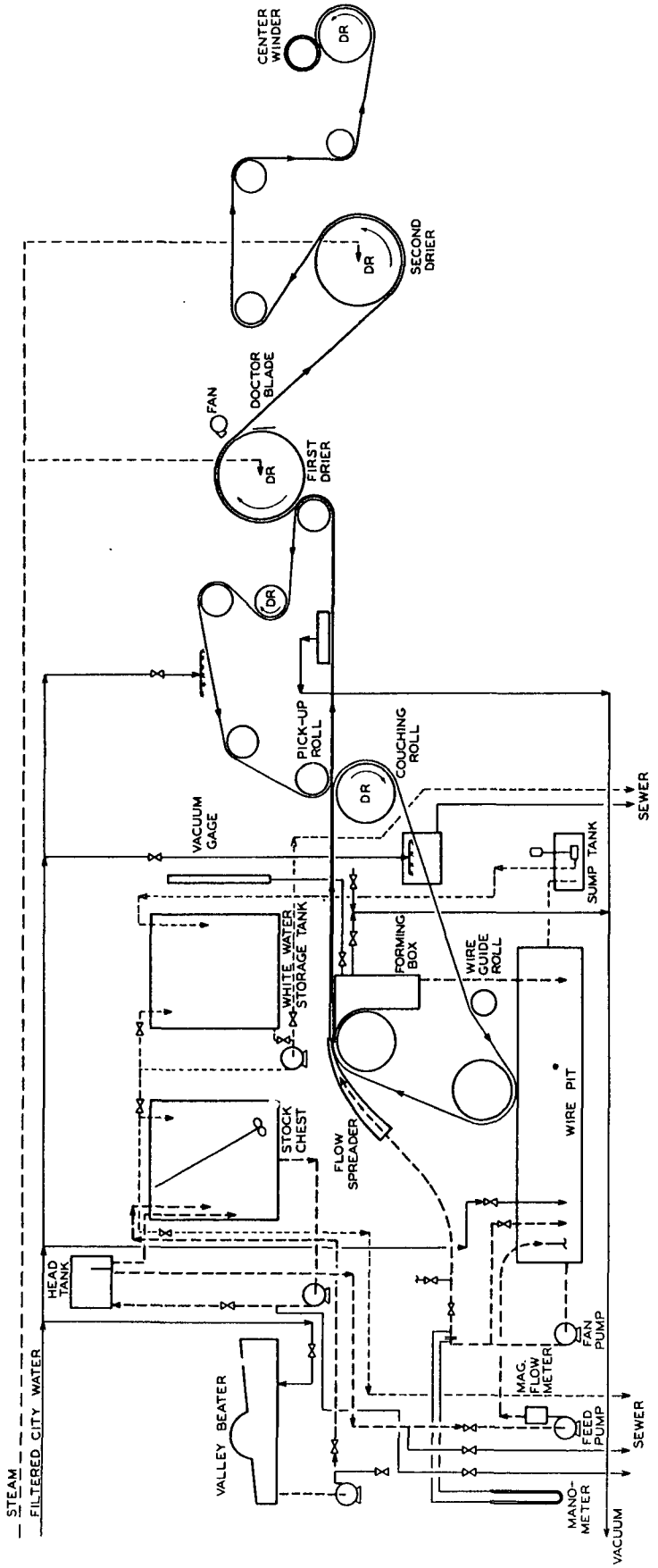

Figure 1. Schematic Flow-Diagram of the IPC Web Former

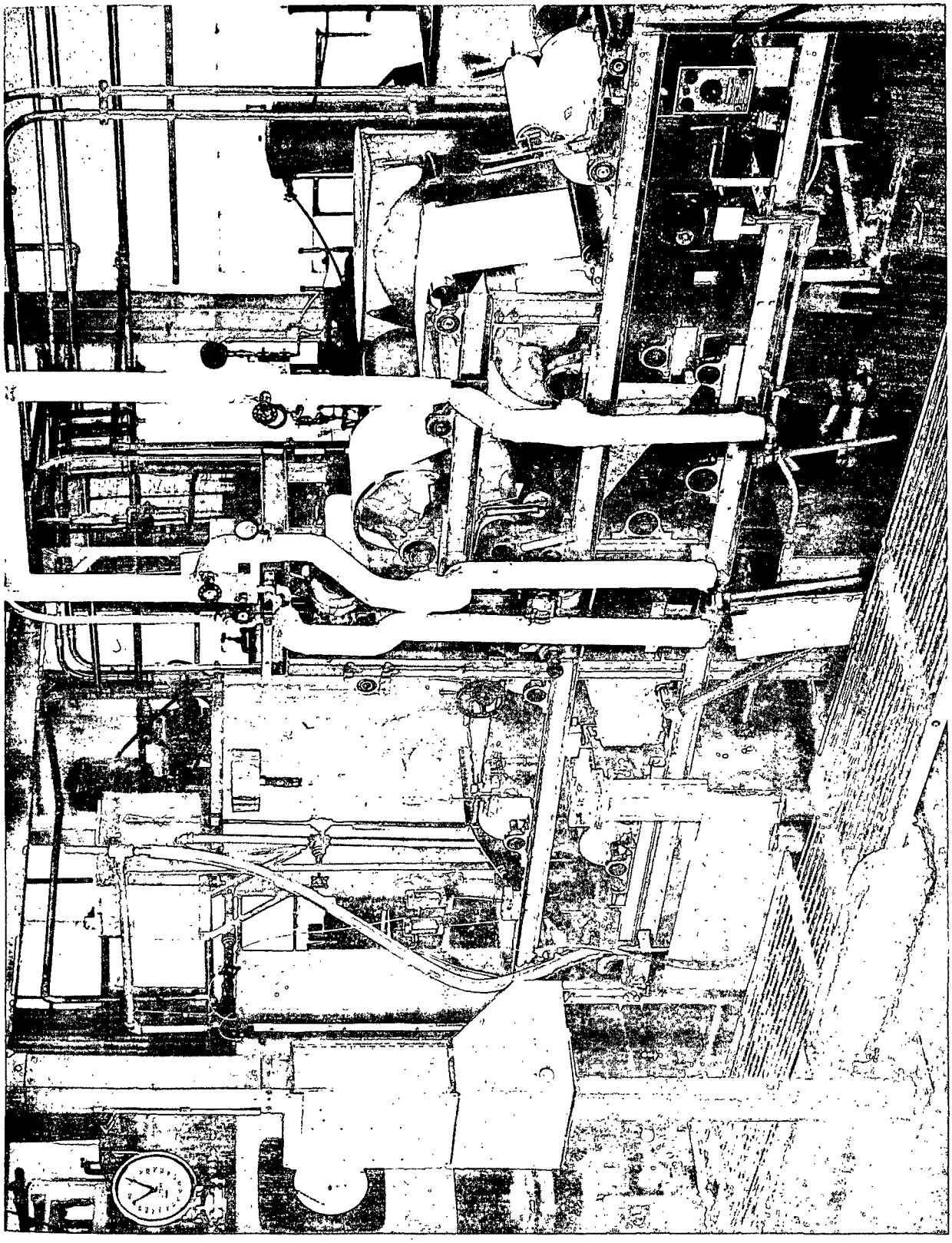

Figure 2. The IPC Web Former

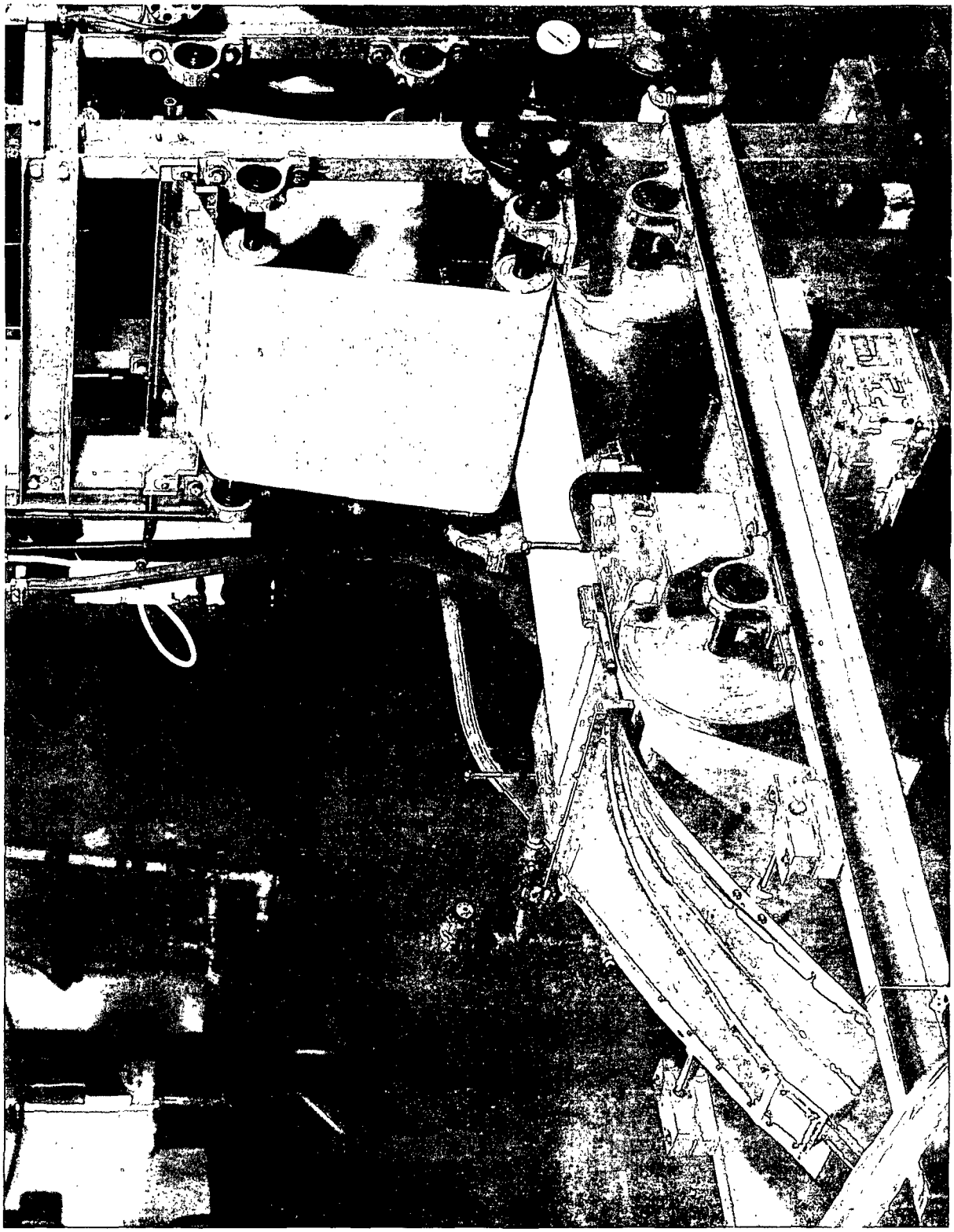

Figure 3. The Sheet-Forming Section of the IPC Web Former

completed. At the end of the aging period, the xanthide (average age 19 min.) was poured into the stock chest and blended in for 5 minutes.

On the basis of a minimum loss during processing, 10 g. xanthide starch were added per 100 grams of fiber (181.6 g. xanthate starch or 0.4 lb. per 4.0 lb. fiber).

The data pertaining to the operation of the web former are shown in Table I.

Unaged starch xanthide (Run No. II) appears to increase the drainage rate on the wire as indicated by the lower vacuum on the forming box. Similar behavior has been observed on the pilot machine in Peoria. Aging the xanthide for 15 minutes does not produce this effect.

Both xanthide runs produced a serious sticking problem on the single drier drum. It was necessary to apply sodium oleate continuously to the drier surface to release the web. This was only partially successful since the machine glazed surface was badly roughened by lifted fibers. Toward the end of Run No. III, Ivory soap was added to the stock. This treatment was effective in eliminating drier sticking but it also reduced the wet and dry tensile strength of the paper. This result (Run No. IIIS) is shown in Table II, along with the results of the physical tests on the three main sets of paper samples.

Both xanthide treatments produced paper having improved wet and dry strength properties as compared with the control. Paper made with xanthide added directly to the fiber as produced is superior to the aged xanthide specimens only in respect to wet tensile strength and in xanthide retention. On the basis of these data, it does not appear that aging dilute starch xanthide preparations for 15 minutes seriously affects the properties imparted to paper products.

TABLE I

WEB-FORMER CONDITIONS

Run number	(I)	(II)	(III)
Xanthide, 10 g./100 g. fiber	None	Unaged	Aged
Stock freeness, ml. S.-R.	710	700	700
Stock consistency, xanthide addition, %	--	1.7	1.6
Stock consistency for run, %	0.05	0.05	0.05
Xanthide, pH	--	5.3	4.9
Stock pH, ready to run	5.3	5.1	5.3
Wire pit pH, end of run	5.7	5.7	5.7
Wire speed, ft./min.	10.7	9.5	9.0
Stock feed, gal./min.	3.9	4.0	4.0
Inlet feed, gal./min.	14.2	12.0	12.5
Forming vacuum, in. water	27	17	25.5
Forming length, cm.	18.5	18.0	18.0

A standard 72 x 56 semitwill bronze wire was used in all three runs.

TABLE II

PHYSICAL CHARACTERISTICS OF PAPER MADE ON IPC WEB FORMER

Run Number Ex Situ Xanthide	(Number of Tests)	I None	II Unaged	III Aged	III S Aged and Ivory Soap
Basis weight (25 x 40/500), lb./ream, g./m.^{2}	(1)	48.0 67.5	52.4 73.7	47.8 67.2	
Apparent density, lb./ream/mil	(7)	10.2	11.1	9.8	
Bursting strength, points/100 lb.	(5)	96	95	111	
Instron dry tensile M.D., lb./in., kg./15 mm. breaking length, m.	(5)	33.1 8.87 8764	38.1 10.20 9231	38.7 10.37 10285	17.7
Schopper wet tensile M.D., lb./in. (30-min. soak) kg./15 mm. breaking length, m.	(5)	1.2 0.32 316	5.2 1.39 1258	4.2 1.13 1121	3.6
Schopper wet tensile M.D., lb./in. (16-hr. soak) kg./15 mm. breaking length, m.	(5)	1.2 0.32 316	5.3 1.42 1285	4.5 1.31 1299	
M.I.T. fold	(10)	2340	3530	4720	
Fluorescence size time, sec.		Instantaneous			
Opacity (felt side)	(5)	75.8	70.7	71.6	
Standard brightness	(5)	84.5	81.4	82.2	
Xanthide, g./100 g. sheet	(2)	--	3.4	2.9	
Retention efficiency, %	--	--	35.2	29.9	

EFFECT OF XANTHIDE DISTRIBUTION IN HANDSHEETS

The xanthide aging experiment (1), mentioned in the preceding section, was the basis for another line of investigation. Even though laboratory preparations of ex situ starch xanthide have a useful storage life of 5 to 60 minutes, further aging does change its behavior in handsheets. As the storage time is increased from 1 to 4 hours, and finally to 24 hours, retention efficiency increases while wet and dry tensile strenghs decrease. This anomaly was investigated further by viewing iodine-stained specimens under the microscope.

The paper made with xanthide aged 5 or 60 minutes stains uniformly with iodine--potassium iodide solutions while the 4- and 24-hour samples have a speckled appearance. Under relatively low power magnification the 5- and 60-minute specimens show uniform coloration of the fibers (Fig. 4 and 5). By raising and lowering the focus of the microscope the mottled appearance of these two figures is seen to be due to areas where greater numbers of fiber segments come between the light source and the viewer. A similar mottled effect is seen in the control (Fig. 6) which contains no starch xanthide.

The speckled appearance of the 4- and 24-hour samples is due to lumps of xanthide mixed in with the fibers (Fig. 7 and 8). These lumps appear, in some cases, to be clusters of smaller xanthide particles. The intensity of fiber staining decreases as the xanthide is aged beyond one hour. At 24 hours, the fiber color is barely changed by staining with iodine, even though this is not obvious when Fig. 7 and 8 are compared. Qualitatively, the color intensity of the stained fibers parallels the wet tensile strength of the paper (Table III). That is, the darker the fiber color produced by iodine staining the higher the wet tensile strength.

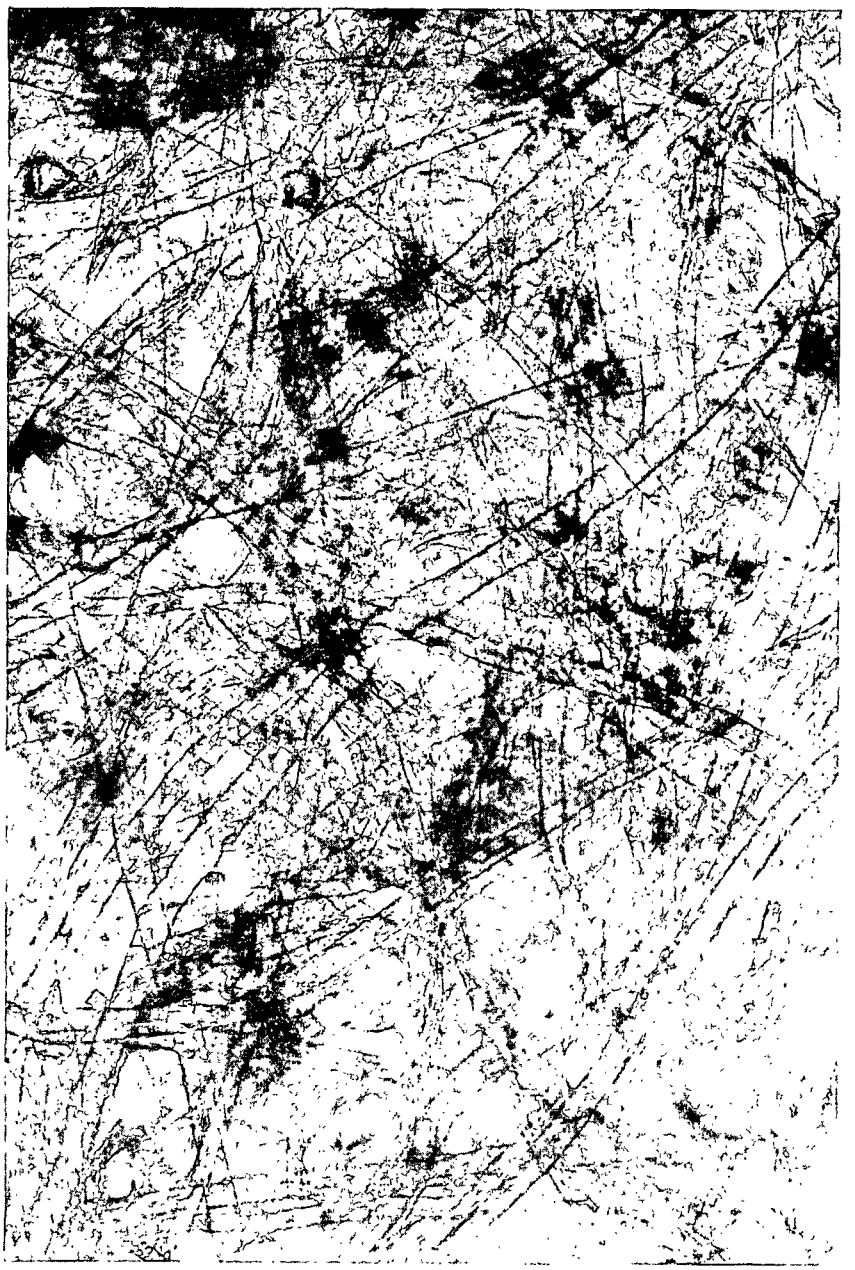

Figure 4. Xanthide Age = 5 Min.
(I_2-KI Stain, 115X)

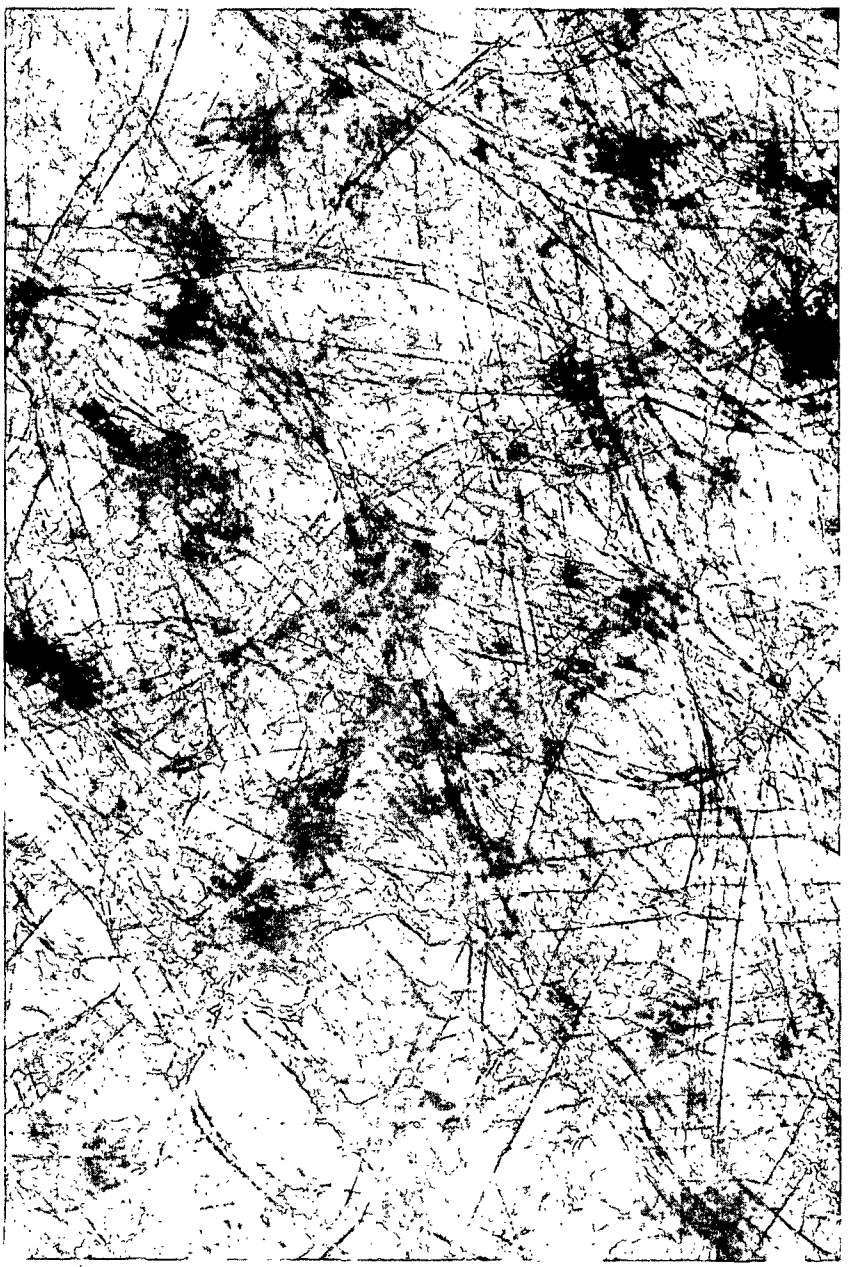

Figure 5. Xanthide Age = 60 Min.
(I_2-KI Stain, 115X)

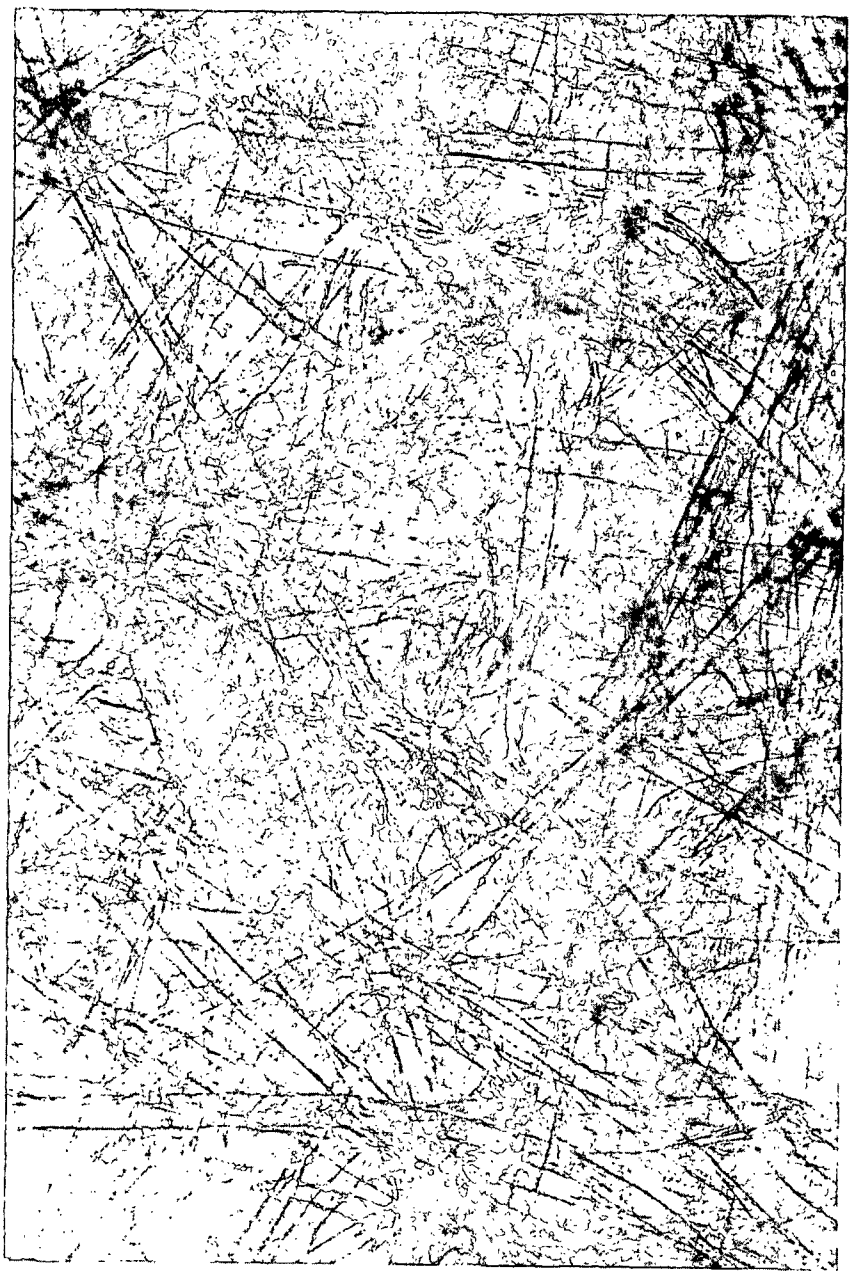

Figure 6. Control Sheet with No Xanthide Added
(I_2-KI Stain, 115X)

Figure 7. Xanthide Age = 240 Min.
(I_2-KI Stain, 115X)

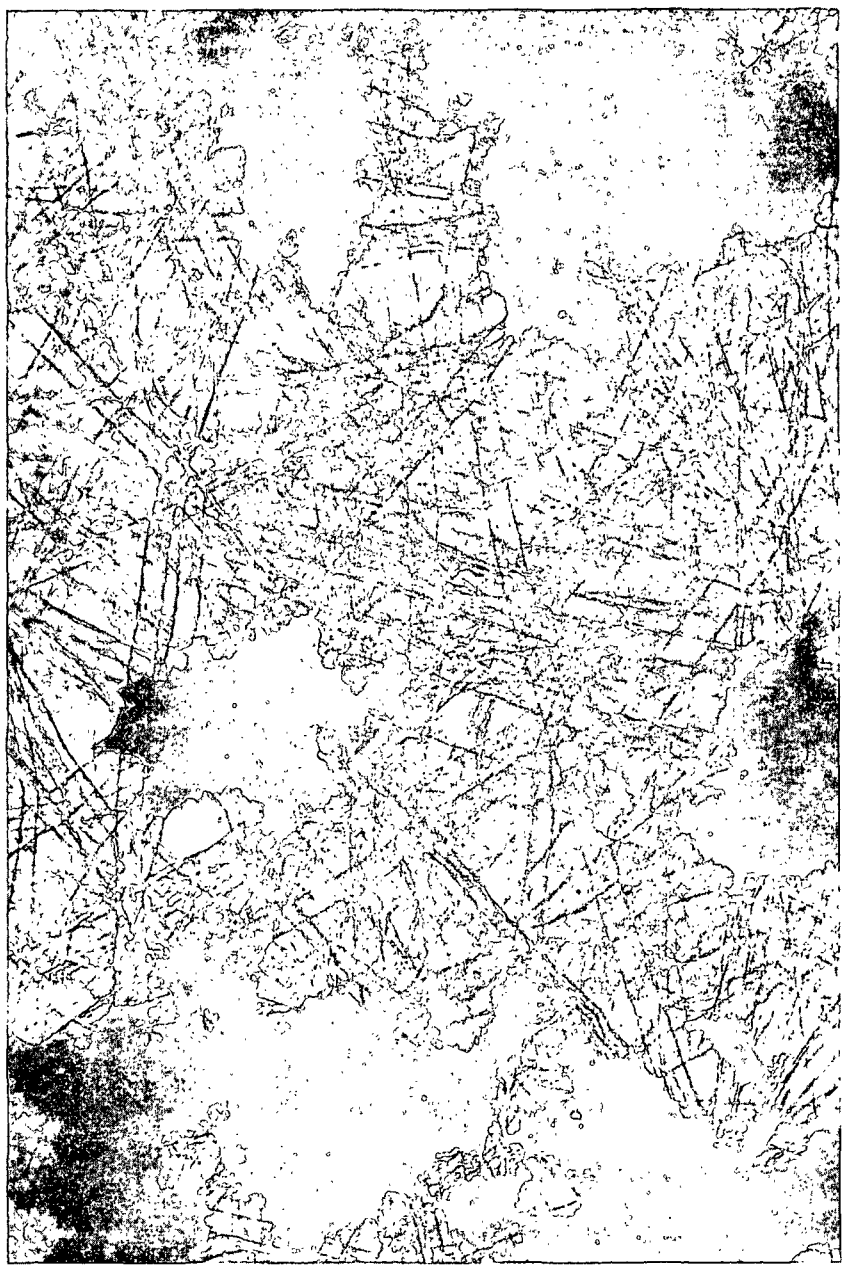

Figure 8. Xanthide Age = 1440 Min.
(I_2-KI Stain, 115X)

TABLE III

EFFECT OF AGE OF EX SITU XANTHIDE PREPARATION UPON HANDSHEETS

(Taken from Report Six)

Preparation No.	Xanthide Age When Used, min.	Xanthide Retained, g./100 g. fiber	Retention Efficiency, %	Instron Dry Tensile, lb./in.	Instron Wet Tensile[a], lb./in.
I	5	1.05	23.3	30.9	3.7
	60	1.42	31.5	31.5	3.4
	240	1.75	38.9	27.5	2.2
	1440	2.32	51.3	24.0	1.3
Blank	--	--	--	20.7	0.7

[a] Soaked 30 minutes in distilled water at 73°F.

Viewing at higher magnification reveals that fibers teased from the paper have roughened surfaces. This surface roughness is not in line with having beaten the bleached softwood kraft fiber only to 700 ml. Schopper-Riegler freeness before removing the fines in a Bauer-McNett classifier. It is believed the roughened surfaces as seen in Fig. 9 and 10 are due to a layer of starch xanthide. The control, Fig. 11, does not display quite the same degree of roughness and the bordered pits (rows of little doughnuts) on the fibers are easily seen. Very few bordered pits are visible on the fibers treated with xanthide 5 and 60 minutes old. It is presumed they are covered up by starch xanthide.

The iodine stain (3) does not produce a color deep enough at this magnification to be useful in locating starch xanthide on the fiber (Fig. 10). Graff's "C" stain (3), which contains aluminum, zinc, and calcium chlorides, as well as potassium iodide and iodine, produces a much deeper color with starch xanthide (Fig. 9). The color produced by the fiber itself (3) is much less intense, as

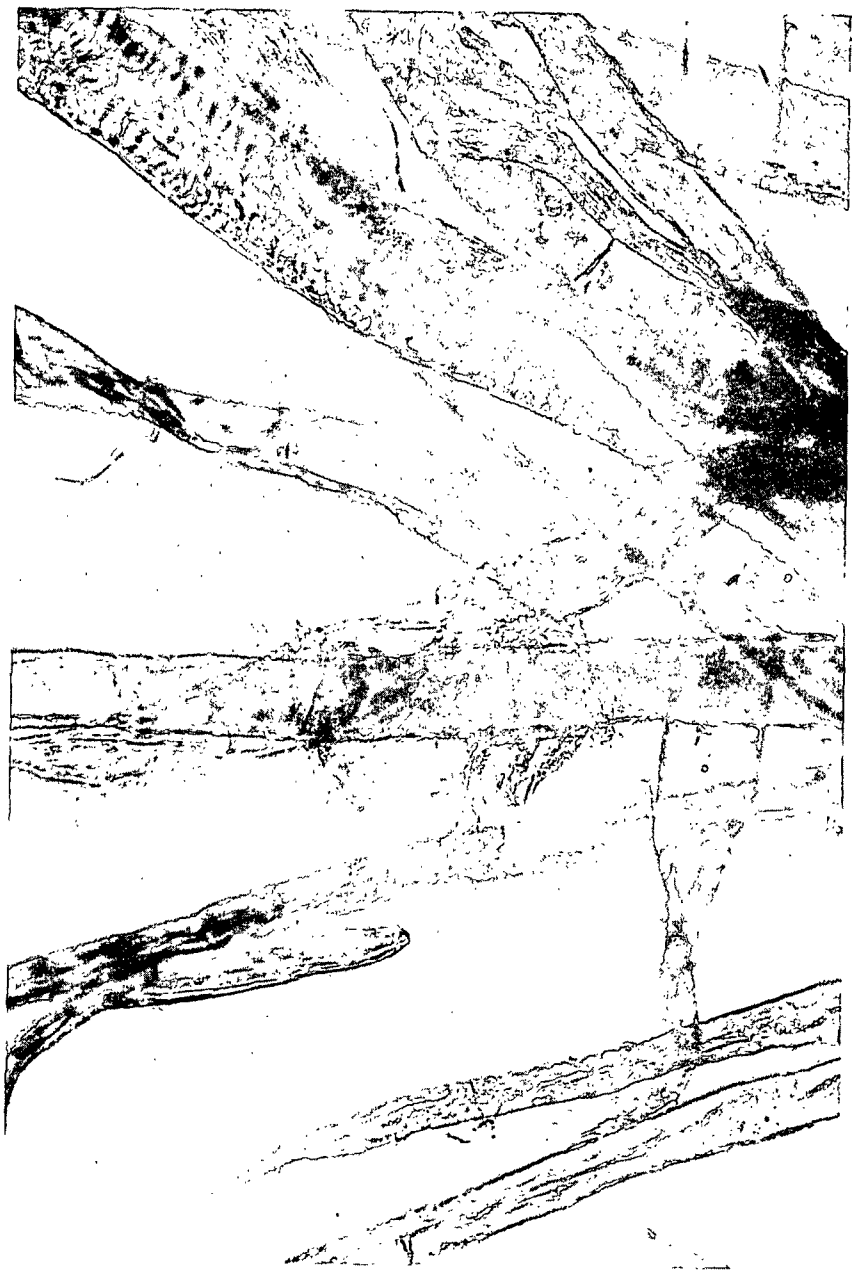

Figure 9. Xanthide Age = 5 Min.
("C" Stain, 300X)

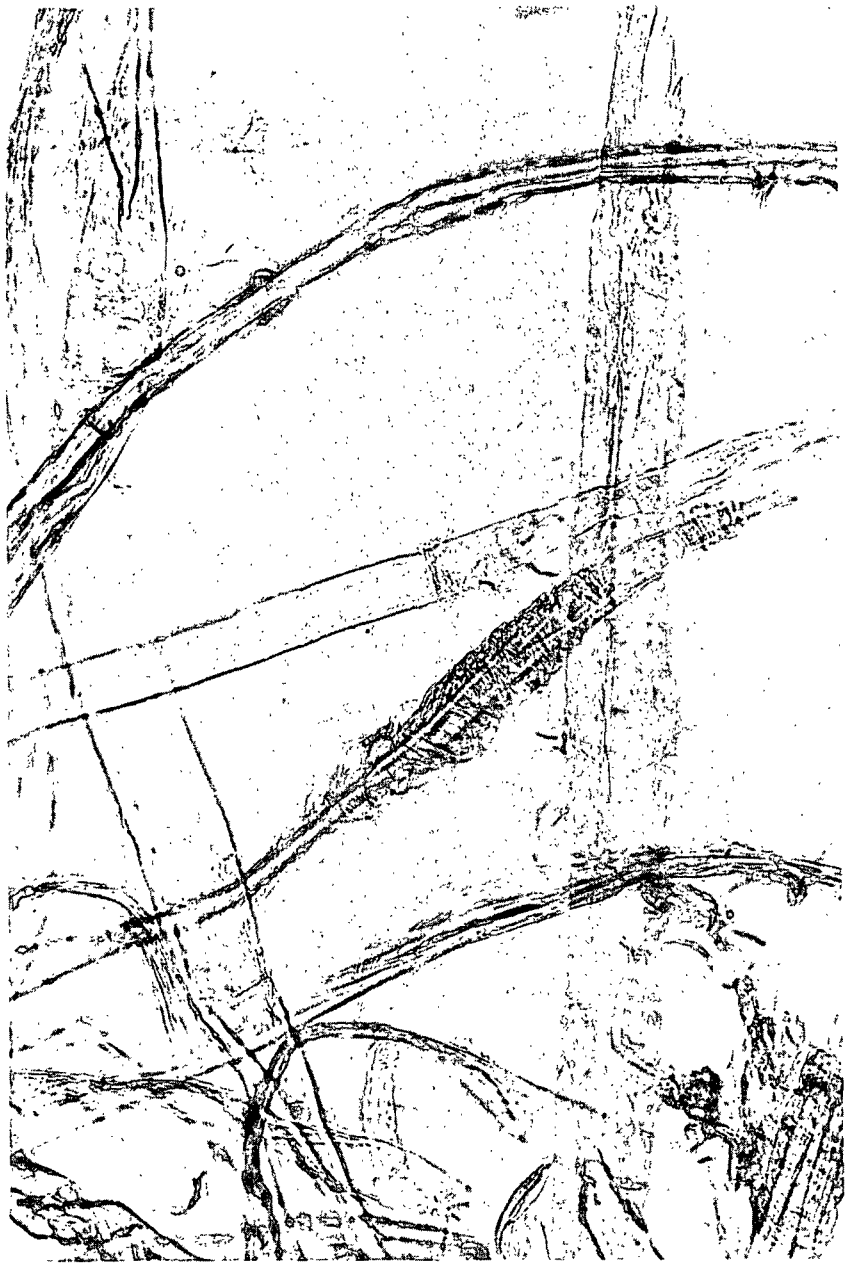

Figure 10. Xanthide Age = 60 Min.
(I_2-KI Stain, 300X)

Figure 11. Control Sheet Containing No Starch Xanthide
("C" Stain, 300X)

may be seen by comparing Fig. 9 and 11. The stain accepted by the fibers in Fig. 9 shows the xanthide to be well distributed on the fiber. In the center of the picture the xanthide deposit either resembles the fibrils from the primary cell wall damaged during beating or it is intimately combined with the fibrils. The degree of fibrillation, in Fig. 9 and 10, is so much greater than in the control (Fig. 11), that at least part must be starch xanthide--perhaps from changing the xanthide layer during removal of the fibers from the paper.

In Fig. 12, a small xanthide particle is viewed at 525 diameters in a specimen from the 4-hour aged xanthide treatment. This particle is about the diameter of the fiber to which it is attached. It is not likely that xanthide retained in this form contributes to the strength of the paper. Just below the particle, a bordered pit is visible. Since these pits occur in rows (Fig. 11) it is likely that there is a layer of starch xanthide which is covering up the rest of the array.

The example of the 24-hour xanthide treatment seen in Fig. 13 has a rather dense xanthide particle which appears to have cemented several fibers together. Such a fiber bond will be effective only if other particles happen to unite the ends of the fibers in the cluster to other fibers. The easily distinguishable bordered pits in Fig. 13 indicate very little xanthide, if any, is deposited on the fiber surface.

The particles seen in Fig. 7, 8, 12, and 13 probably are shaped like corn flakes. Cross-section viewing of the 4-hour xanthide specimen (Fig. 14) shows streaks rather than globules of starch xanthide. It may be that globular particles of xanthide were flattened when the handsheets were formed and dried.

Figure 12. Xanthide Age = 240 Min.
(I_2-KI Stain, 525X)

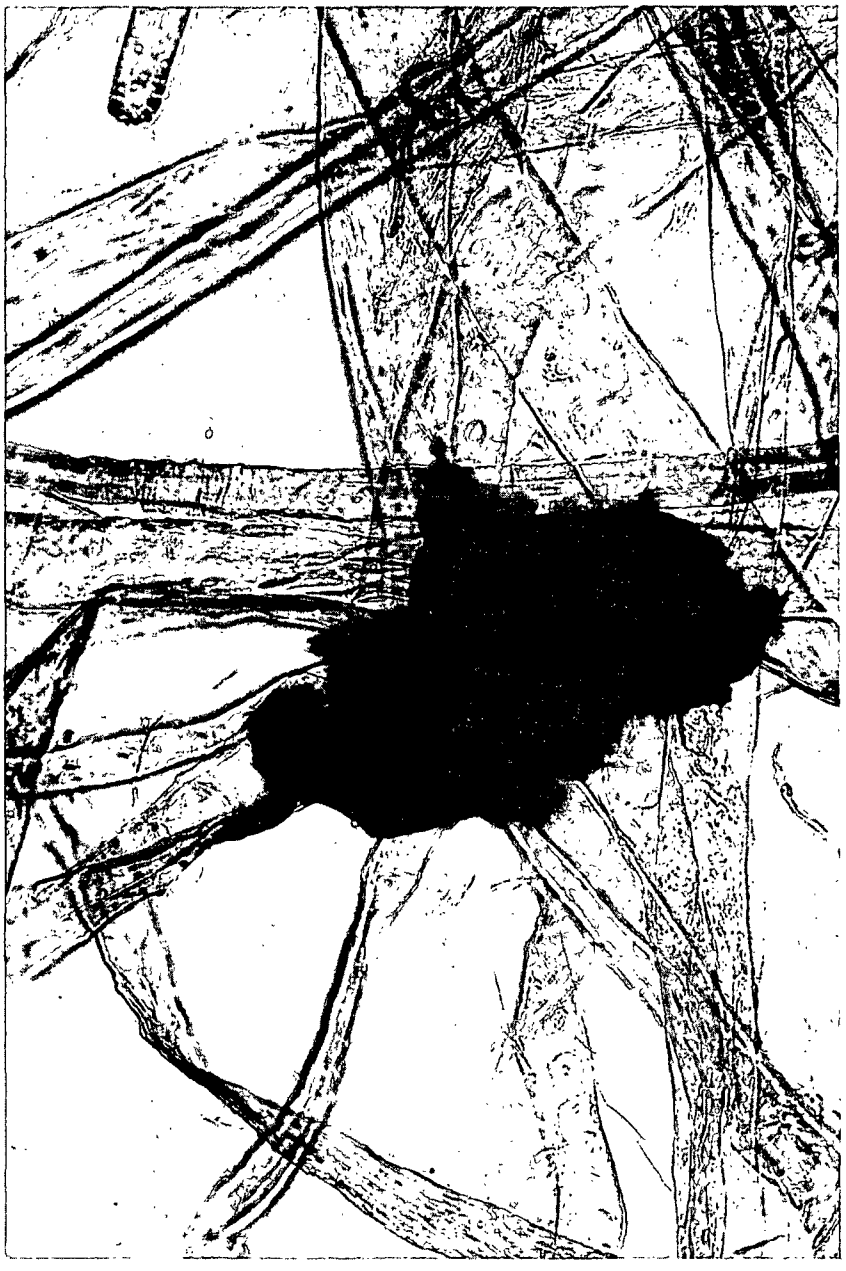

Figure 13. Xanthide Age = 1440 Min.
(I_2-KI Stain, 300X)

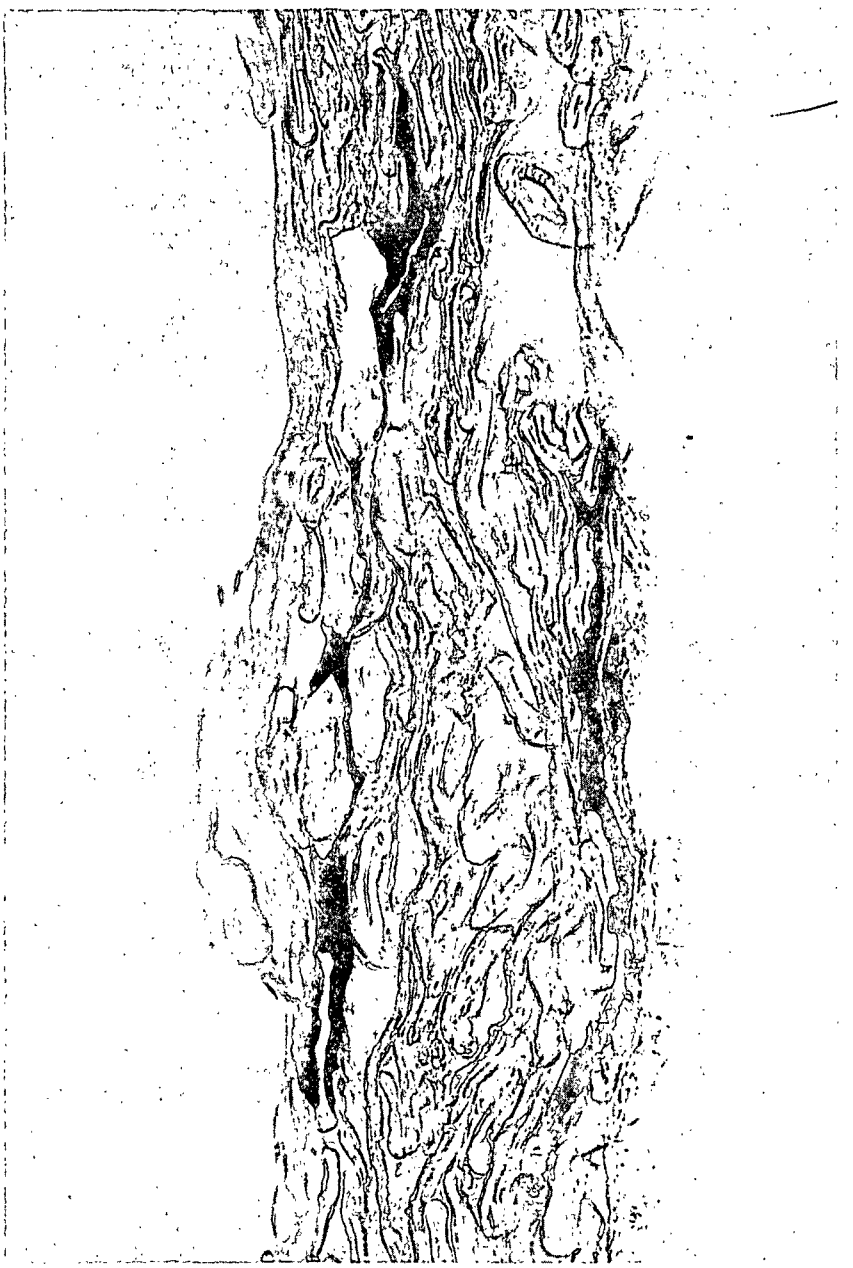

Figure 14. Xanthide Age = 240 Min.
Cross-Sectioned After Imbedding in
Butyl Methacrylate (I_2-KI Stain, 485X)

Figure 15 shows a fiber treated with ex situ starch xanthide which had been aged 60 minutes at 20°C. A sleeve of material which may or may not be starch xanthide is partially removed from the damaged end of the fiber. The iodine--potassium iodide stain used does not produce enough color at this magnification to give positive identification. However, the surface textures of the sleeve and the exposed fiber suggest the sleeve is something other than the primary cell wall. What appears to be a bordered pit is visible in the sleeve segment removed from the fiber. This may be a replica of a pit in the xanthide or the sleeve may be the primary wall itself but reinforced with starch xanthide.

The relationship of the appearance of the paper and the individual fibers to the physical and chemical data (Table III) suggests that coagulation of starch xanthide should be avoided whether cross-linking is done in the presence or absence of the wood fibers. The large particles of coagulum, formed in the 4- and 24-hour samples, apparently contribute very little to the wet tensile strength, even though they apparently improve retention.

It is interesting that few particles are smaller than the diameter of the fibers (see Fig. 4 and 5). This may indicate that smaller particles are not retained by entrapment when the sheet is formed or that xanthide aggregates approaching the size of the fiber are competing with the fiber for smaller units of dispersed starch xanthide. Such an aggregate should offer similar changes in surface free energy as the fiber. This suggests that the starch xanthide should be produced under conditions which form xanthide units several times smaller in diameter than the fiber being used.

Figure 15. Photomicrograph Showing a Sleeve of Material, Possibly Starch Xanthide, Partially Removed From a Fiber Treated with ex situ Xanthide Aged 60 Minutes (I_2-KI Stain, 300X)

XANTHIDE RETENTION RATES AS FUNCTIONS OF TEMPERATURE AND ALUM CONCENTRATION

The effect of 2×10^{-5}, 2×10^{-4}, and 2×10^{-3} molar aluminum sulfate octadeca-hydrate upon ex situ starch xanthide retention rates was studied at 15.0, 25.0, and 35.0°C. Wet and dry tensile strengths were also observed as functions of these variables.

Fines-free Rayonier bleached softwood kraft fibers (17.14 g. dry basis, beaten to 700-ml. S.-R. freeness before classification) were dispersed in 2 liters deionized water for 300 counts in a British disintegrator and made up to 10 liters with more deionized water (resistance = $2.0 \pm 0.5 \times 10^{6}$ ohms). Aluminum sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$, Reagent Grade) solution was added to the stock to give a total of 15.6, 1.56, or 0.156 g. alum. These quantities of alum yield molar concentrations of 2×10^{-3}, 2×10^{-4}, and 2×10^{-5} when 1400 ml. of the fiber suspension is mixed with 200 ml. xanthide preparation. The fiber suspension was tempered to about the temperature of the experiment before six, 1400-ml. portions were placed in 2-quart fruit jars and brought to the desired temperature in a water bath.

The ex situ starch xanthide was produced fresh for each rate experiment according to the procedure developed in Report Six. Starch xanthate (2.50 g. as starch) from Run No. 510 (9.4 g. starch/100 g. solution, D.S. = 0.13) was diluted with 1475-ml. deionized water, equilibrated to 20.0°C. in a water bath and acidified with 20% acetic acid to pH 5.0-5.2. Potassium iodide (5 ml., 5%) was added during the 5-minute interval after acidification. Acidified 1% sodium hypochlorite (20% HOAc to pH 5.0-5.2) was then added to the xanthate to produce a permanent blue color which indicates the completion of the xanthide reaction. The slightly opalescent, blue xanthide preparation (0.17%) was diluted to 2.000 liters (0.125% xanthide starch) and maintained at 20.0°C. for 15 minutes.

Two-hundred milliliter portions of the xanthide preparation (0.25 g.) were added to the 1400-ml. portions of fiber suspension (2.40 g.) at the desired temperature. For 1/4, 1/2, and 1-minute sorption, the preparations were blended manually while small laboratory mechanical stirrers were used to mix the 4, 16, and 32-minute preparations. The contact interval was terminated by pouring the treated fiber into 7 l. deionized water in an 8 x 8-inch Noble and Wood sheet mold and forming a sheet. About 6 seconds elapsed while the deckle box was draining.

To compensate for heating or cooling the sorption mixture by adding 200 ml. of 20.0°C. xanthide solution, the fiber suspension was equilibrated to 14.3, 25.7, or 37.1°C. before the xanthide was added. This combination of temperatures produces the stated sorption temperature of 15.0, 25.0, and 35.0°C. These temperatures were then maintained in a regulated water bath during the longer contact periods.

As in the previous report, the handsheets formed in the Noble and Wood machine were not wet-pressed. This introduces more variation in the tests for tensile strength but does permit removing the handsheets from the blotters when xanthide retention is high.

The handsheets were dried on a steam drum for 3 minutes between blotters, couch blotter up, and then for 3 more minutes with both blotters removed.

The results of testing the handsheets are presented in Tables IV, V, and VI.

The analyses were finished after the manuscript was prepared. Because of this, interpretation of the data will be continued in the next report.

TABLE IV

XANTHIDE SORPTION AT 15.0°C.

Set Number	Xanthide pH	Alum Concentration, molar	Stock, pH	Contact Time, min.	Dry[a] Instron Tensile, lb./in.	Wet[b] Instron Tensile, lb./in.	Glucan, %	Xanthide (Sample-Blank[c]), %	Xanthide, g./ 100 g. fiber	Retention Efficiency, %
(No. Tests)					(2)	(2)	(2)			
15-3 (5)	5.0	2×10^{-3}	5.0 (NaOH)	1/4	--	--	6.45	1.43	1.45	13.9
				1/2	--	--	6.48	1.46	1.48	14.2
				1	--	--	6.71	1.69	1.72	16.5
				4	--	--	7.20	2.18	2.23	21.4
				16	--	--	7.80	2.78	2.86	27.5
				32	--	--	8.27	3.25	3.36	32.3
				Blank	--	--	4.98[c]	--	--	--
15-3	5.1	2×10^{-3}	3.3	1/4	20.8	1.41	6.68	1.66	1.69	16.2
				1/2	18.6	1.24	7.02	2.00	2.04	19.6
				1	23.0	1.88	7.28	2.26	2.31	22.2
				4	24.2	2.78	8.16	3.14	3.24	31.1
				16	23.9	3.36	9.34	4.32	4.52	43.4
				32	27.4	3.90	8.84	3.82	3.97	38.2
15-4	4.9	2×10^{-4}	4.0	1/4	16.0	0.75	5.78	0.76	0.76	7.3
				1/2	16.2	0.85	6.10	1.08	1.09	10.5
				1	17.6	1.10	6.63	1.61	1.64	15.8
				4	23.2	1.82	6.81	1.79	1.82	17.5
				16	23.6	2.44	7.37	2.35	2.41	23.2
				32	24.3	1.98	7.56	2.54	2.61	25.1
				Blank	13.0	0.43	5.06[c]	--	--	--
15-5	5.2	2×10^{-5}	4.6	1/4	13.4	0.54	5.26	0.24	0.24	2.3
				1/2	12.7	0.44	5.34	0.32	0.32	3.1
				1	13.1	0.52	4.87	(-0.15)	--	--
				4	14.6	0.68	5.37	0.35	0.35	3.4
				16	15.7	0.72	5.98	0.96	0.97	9.3
				32	17.6	0.78	6.29	1.27	1.29	12.4

[a] 50% R.H. at 73°F.

[b] Soaked overnight in distilled water at 73°F.

[c] Average = 5.02.

TABLE V

XANTHIDE SORPTION AT 25.0°C.

Set Number	Xanthide pH	Alum Concentration, molar	Stock, pH	Contact Time, min.	Dry[a] Instron Tensile, lb./in.	Wet[b] Instron Tensile, lb./in.	Glucan, %	Xanthide (Sample-Blank), %	Xanthide, g./ 100 g. fiber	Retention Efficiency, %
(No. Tests)					(2)	(2)	(2)			
25-3	5.3	2×10^{-3}	3.4	1/4	17.2	1.12	6.50	1.19	1.20	11.5
				1/2	19.0	1.31	6.43	1.12	1.13	10.9
				1	19.6	1.36	6.62	1.31	1.33	12.8
				4	22.1	2.04	7.38	2.07	2.11	20.3
				16	21.2	2.96	8.45	3.14	3.24	31.1
				32	24.9	3.30	9.24	3.93	4.09	39.3
				Blank	10.5	0.35	5.31	--	--	--
25-4	5.1	2×10^{-4}	4.1	1/4	15.8	0.88	6.05	0.74	0.74	7.1
				1/2	17.2	1.08	6.40	1.09	1.10	10.6
				1	15.2	0.92	6.64	1.33	1.35	13.0
				4	21.2	1.68	7.20	1.99	2.03	19.5
				16	21.2	2.34	7.47	2.16	2.21	21.2
				32	25.8	2.76	7.45	2.14	2.19	21.0
25-5	5.1	2×10^{-5}	4.6	1/4	12.9	0.48	5.60	0.29	0.29	2.8
				1/2	13.6	0.54	6.01	0.70	0.70	6.7
				1	13.6	0.55	5.77	0.46	0.46	4.4
				4	16.2	0.79	5.78	0.47	0.47	4.5
				16	15.7	0.98	6.20	0.89	0.90	8.6
				32	15.0	0.88	6.02	0.71	0.72	6.9

[a] 50% R.H., 73°F.

[b] Soaked overnight in distilled water at 73°F.

TABLE VI

XANTHIDE SORPTION AT 35.0°C.

Set Number	Xanthide pH	Alum Concentration, molar	Stock, pH	Contact Time, min.	Dry[a] Instron Tensile, lb./in.	Wet[b] Instron Tensile, lb./in.	Glucan, %	Xanthide, %	Xanthide, g./ 100 g. fiber	Retention Efficiency, %
(No. Tests)					(2)	(2)	(2)			
35-3		2×10^{-3}		1/4	19.2	1.18	6.69	1.05	1.06	10.2
				1/2	18.3	1.32	6.86	1.22	1.24	11.9
				1	18.6	1.42	6.98	1.34	1.36	13.1
				4	24.3	2.12	7.34	1.70	1.73	16.6
				16	21.6	1.98	7.78	2.14	2.19	21.0
				32	24.2	2.26	7.96	2.32	2.38	22.9
				Blank	10.8	0.39	5.64	--	--	--
35-4		2×10^{-4}		1/4	17.6	1.24	6.58	0.78	0.79	7.6
				1/2	18.4	1.40	6.76	0.96	0.97	9.3
				1	22.3	1.71	7.07	1.27	1.29	12.4
				4	22.9	2.73	7.09	1.29	1.31	12.6
				16	23.6	2.61	7.34	1.54	1.57	15.1
				32	24.4	2.71	7.74	1.94	1.98	19.0
				Blank	13.2	0.36	5.80	--	--	--
35-5		2×10^{-5}		1/4	13.0	0.50	5.86	0.14	0.14	1.3
				1/2	13.4	0.59	5.76	0.04	0.04	0.4
				1	14.2	0.61	5.90	0.18	0.18	1.7
				4	14.3	0.67	5.62	(-0.10)	--	--
				16	16.2	0.69	5.60	(-0.12)	--	--
				32	14.8	0.62	5.62	(-0.10)	--	--
				Blank	11.6	0.38	5.72	--	--	--

[a] 50% R.H., 73°F.

[b] Soaked overnight in distilled water at 73°F.

Figures 16 and 17 show that both xanthide sorption and wet tensile strength follow a linear log-log relationship with the sorption time. Raising the sorption temperature from 15 to 35°C. decreases xanthide sorption and wet tensile strength in 2×10^{-3} molar alum.

In Fig. 18 and 19, the alum concentration has a strong effect upon sorption and wet tensile strength at 15°C. It appears that further evaluation of alum concentration exceeding 2×10^{-3} molar is warranted.

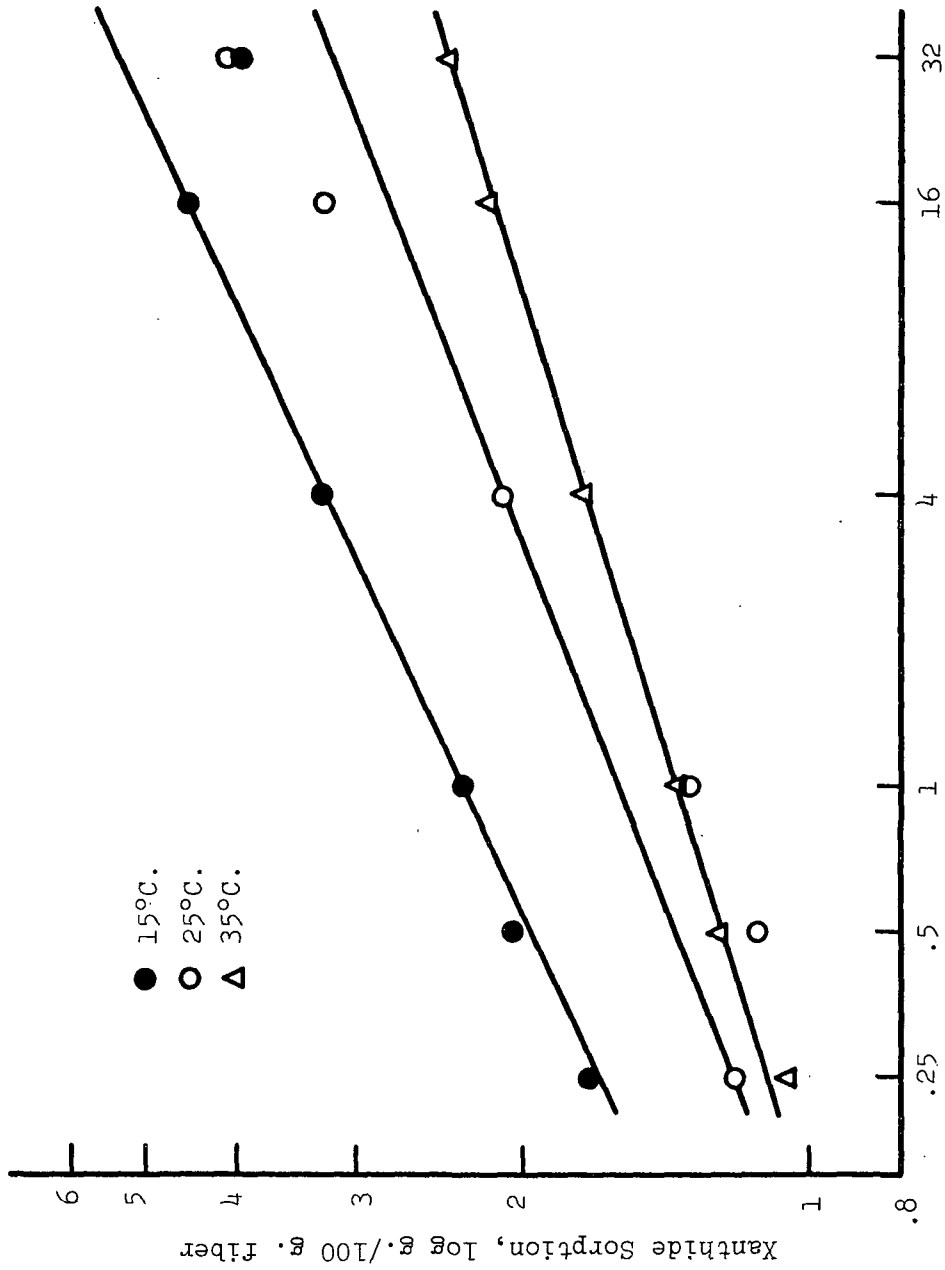

Figure 16. Double Log Plot of Xanthide Sorption vs. Time in 2 x 10^{-3} Molar Alum

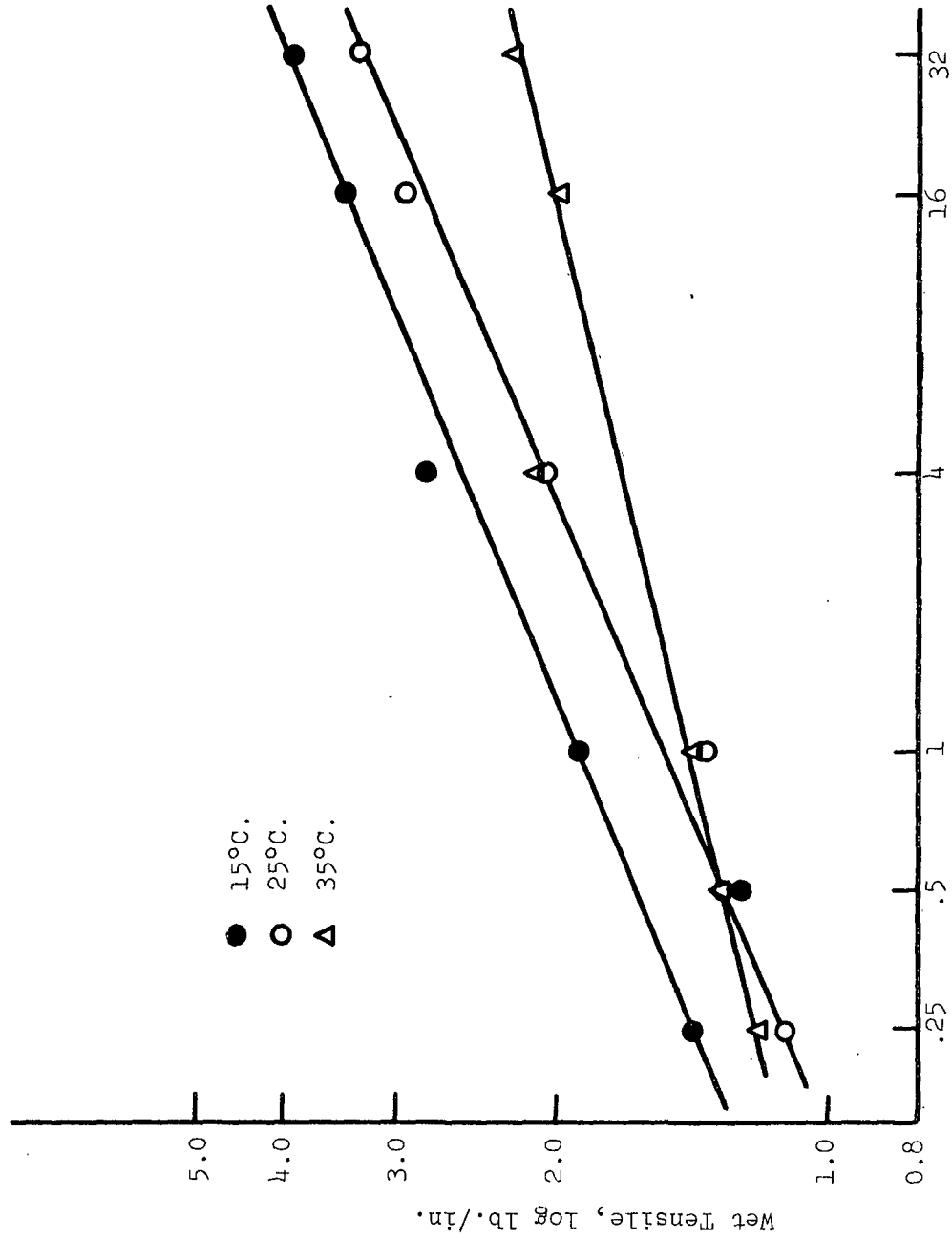

Figure 17. Double Log Plot of Wet Tensile Strength vs. Time in 2×10^{-3} Molar Alum

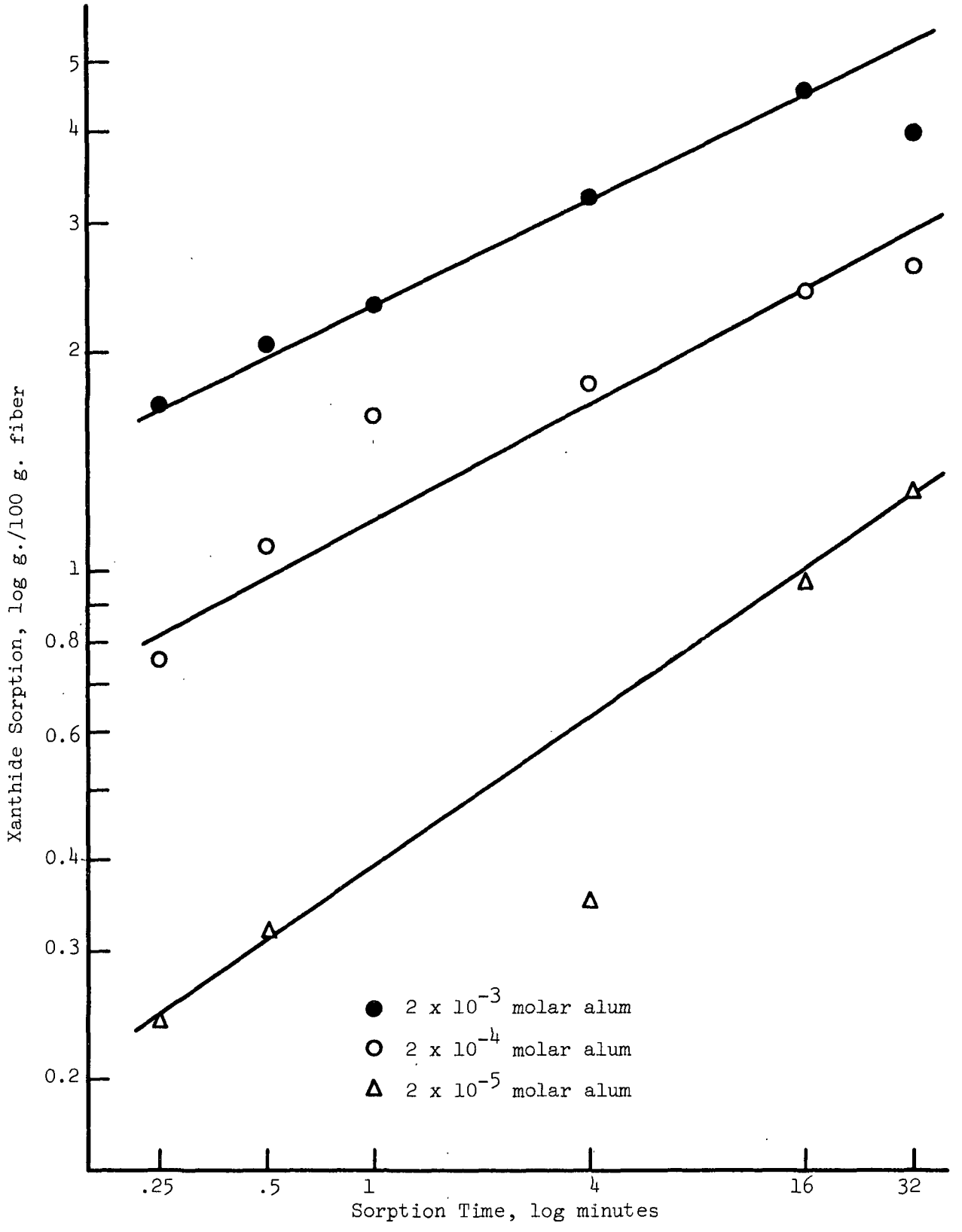

Figure 18. Double Log Plot of Xanthide Sorption vs. Time at 15°C.

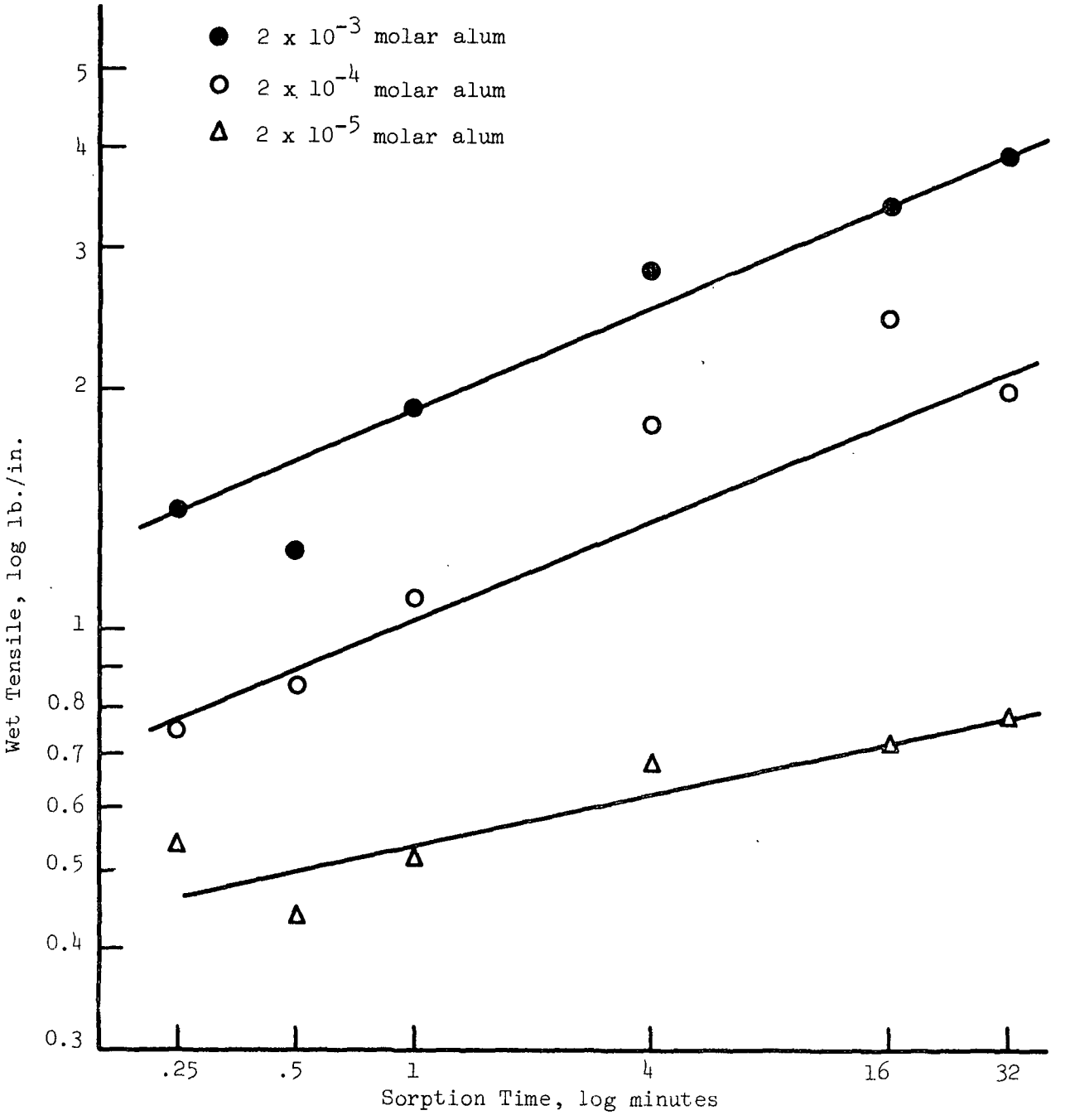

Figure 19. Double Log Plot of Wet Tensile vs. Time at 15°C.

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THE INSTITUTE OF PAPER CHEMISTRY

George Lauterbach
George Lauterbach
Research Associate

Edward J. Jones
Edward J. Jones
Chief, Colloid Chemistry Group

John W. Swanson
John W. Swanson
Chairman, Physical Chemistry Section