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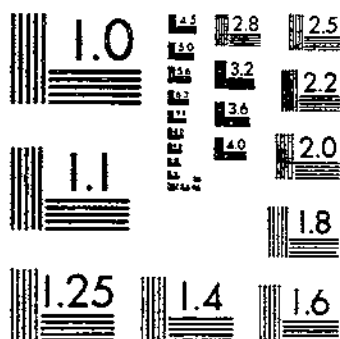
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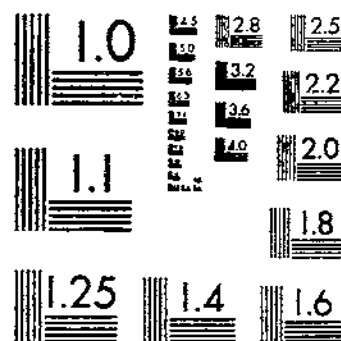
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SOME PHYSICAL PROPERTIES OF STARCH PASTES WHICH AFFECT THEIR STIFFENING
FLURRY, N. S. 1 OF 1**

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MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A



MICROCOPY RESOLUTION TEST CHART
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UNITED STATES DEPARTMENT OF AGRICULTURE
WASHINGTON, D. C.SOME PHYSICAL PROPERTIES OF STARCH PASTES
WHICH AFFECT THEIR STIFFENING
POWER ON FABRICS¹By MARGARET S. FURRY, *Assistant Textile Chemist, Textiles and Clothing Division,
Bureau of Home Economics*

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INTRODUCTION

As part of a series of studies on home laundering, an attempt has been made to evaluate the materials and methods that may be employed in sizing fabrics. In connection with the work on stiffness (17)² attention was directed to those physical properties of starch pastes that seem to influence the stiffness of fabrics sized with the pastes.

Although several investigators (5, 7, 9, 14, 15, 24) have studied the relationship between penetration of starch pastes into yarns and fabrics and the apparent viscosity or consistency of the pastes, there has been no correlation of these facts with any properties of sized fabrics. It is the purpose of this bulletin to point out the correlation of fabric stiffness produced by various starch pastes to the consistency of these pastes and to their penetrating and coating powers on the fabric. Size measurements were taken of both the raw and swollen starch granules, consistency determinations of the starch pastes were made at one concentration and temperature, and the penetrating and coating powers of the starches were determined by examining cross sections of the sized yarns and fabric. These investigations were carried out with potato, corn, wheat, rice, canna, and dasheen starches. Canna and dasheen starches have not heretofore been used in the sizing of textiles, but since the United States Department of Agriculture has been interested in cultivating the plants and in the possible extension of the use of their products, a study of the properties of these starches is being made.

¹ Acknowledgment is made to Ruth E. Elmquist, of the Textiles and Clothing Division, for cooperation in determining the rate of flow of starch pastes; to Virginia Hefty and Eleanor Bowen for general laboratory assistance; to W. H. Herschel and R. Bulkley, of the United States Bureau of Standards, for helpful suggestions on consistency measurements of the starch pastes; and to M. L. Fobart, of the section of Illustrations, United States Department of Agriculture, for taking the photomicrographs used in this bulletin.

² Italic numbers in parentheses refer to Literature Cited, p. 16.

THE STARCHES

Photomicrographs of the starches studied are shown in Plate 1, A to G inclusive. The potato, canna, corn, wheat, and rice starches, according to descriptions given by Reichert (18) are true to type. These were prepared as described by Peterson and Dantzig (17) from Fortuna rice, Kharkof hard-winter wheat, Russet Rural potatoes, and Boone County White corn. The edible-canna starch, which was obtained from J. C. Ripperton of the Hawaii Agricultural Experiment Station, came from canna grown in the Waimea district of Hawaii. It had been extracted according to the method described by Ripperton and Goff (20). The dasheens (*Colecasia esculenta* (L.) Schott) (23) from which the dasheen starch was prepared are a species belonging to the Aroideae and are somewhat similar to the plant popularly known as elephant-ear. The tubers were grown in Florida and were provided through the courtesy of R. A. Young, Bureau of Plant Industry, United States Department of Agriculture.

In extracting the starch from dasheens the tubers were peeled and finely ground. The pulp was put in heavy muslin bags and worked by hand in tubs of distilled water which was changed frequently. The washing was continued until the final wash water remained almost clear. Then the wash waters were combined, and the starch was centrifuged out. After several washings in fresh distilled water and centrifuging, the starch was washed with alcohol, tested for protein with Millon's reagent, ether-washed until free from fat, air-dried at room temperature and put through a 70-mesh sieve. It was necessary to use the centrifuge each time the starch was washed because the dasheen-starch granules are extremely small, and a gummy substance present in the dasheen prevents rapid settling of the granules when suspended in water.

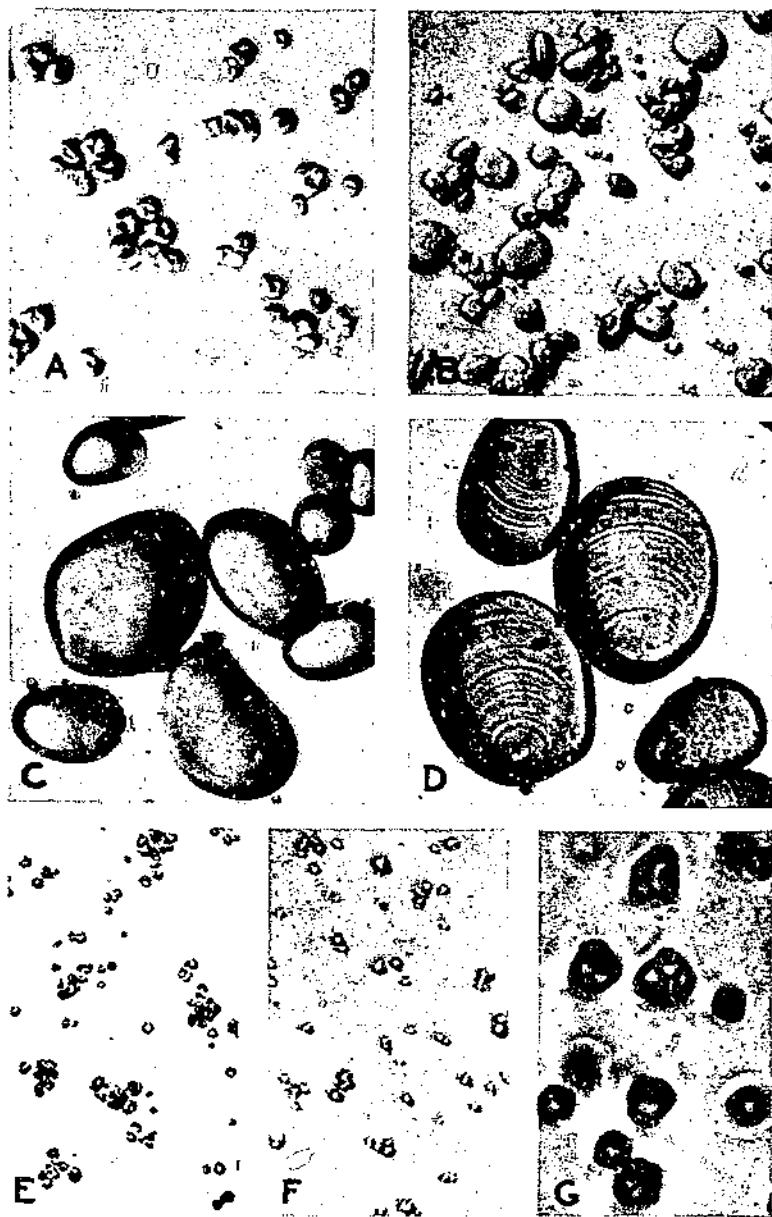
The dasheen-starch granules, which are the smallest starch granules known, occur sometimes in small aggregates but are mostly isolated. The most conspicuous forms appear to be irregular or rounded polygonal shapes. They have distinct, clear-cut, and somewhat concave facets. The hilum is small, fairly distinct, usually situated near the center, and often appears as a smooth hole. The striae are either invisible or indistinct.

SIZE OF THE STARCH GRANULES

In order to describe more fully the starches used in this study, measurements were made of the size of both the raw and the swollen starch granules. The latter are of special interest because most investigators (24) now believe that as starch granules are heated in water to form a paste they swell but do not ordinarily disintegrate or become disrupted, and also because it is generally accepted (1, 9, 21, 22) that the number and size of swollen starch granules in a paste and the consistency of that starch paste are closely related.³

From various observations Alsberg (1) concludes that starch pastes, unless specially treated to disintegrate the particles, are not true colloidal solutions but are suspensions of swollen granules. He also believes that the viscosity of a paste depends in a large part upon the anatomical structure of the swollen granules. He attributes the swelling of the granules to three factors: Rigidity, inherent swelling

³ CHAPMAN, O. W., PROPERTIES OF STARCH WITH RELATION TO TIME OF FORMATION OF STARCH GELS. 1927. [Unpublished thesis. Iowa State Col. Agr. Mechanic Arts, Ames.]



PHOTOMICROGRAPHS OF RAW STARCH GRANULES

A, Corn starch; B, wheat starch; C, potato starch; D, cassia starch; E, rice starch; F, dasheen starch; G, dasheen starch. A-F $\times 250$; G $\times 1,600$.

power of the granule substance, and the relation of the mass of the swelling substance to the surface area of the granule. This third factor, which is the relation of increase of volume of spheres to the increase in surface area, indicates that large starch granules have greater mass of swelling substance exerting pressure on each unit of granular surface than smaller ones. Therefore, as Alsberg states, of two granules having the same rigidity but different diameters, the larger one will swell more readily and probably sooner, as the temperature is raised, than the smaller one. This is because the greater mass of the larger will exert pressure to distend its granule structure before the structure of the smaller one has been sufficiently softened to cause it to swell. It seems, therefore, from Alsberg's work, and Chapman⁴ agrees in his work on starches, that larger granules begin swelling at a lower temperature than smaller ones and that the swelling of the smaller granules is a slower process.

Sprockhoff and Parlow (21) in an effort to find the comparative value of small and large potato-starch granules separated the small from the large and made viscosity measurements on the respective pastes. These authors report that small granules of potato starch, if care has been maintained in the manufacture, show even a higher viscosity than the large granules. It must be remembered, however, as Alsberg (1) and Harrison (9) have pointed out, that potato starch presents an exceptional behavior, in that the granules when heated in water not only swell but gradually fragmentize and disintegrate. There is, therefore, the possibility that the viscosity of the pastes used by Sprockhoff and Parlow was low because the large granules had become disrupted.

Ripperton (19), on studying the physicochemical properties of three samples of edible-canna and two samples of potato starches, measured the differences in the swelling power of the starch granule and the viscosity of their pastes at various concentrations. He concluded that the swelling power and viscosity of these starches are not directly proportional but that there is a direct relationship between the viscosity and the product of the concentration of the paste and the volume or "swell" of the swollen starch granules. He found that potato starch swells more and gives a higher viscosity than canna starch.

Chapman⁴ noticed for rice, corn, wheat, and potato starch pastes of 3 per cent concentration that the temperature of maximum viscosity varied with the average diameter of the respective starch granules.

Taylor (22) on trying to rupture cornstarch granules by grinding the dry grains and shaking and grinding starch pastes, found that complete or partly complete rupturing of the granules is not easily accomplished. From microscopical examination, viscosity measurements, and determinations of the amount of the two constituents, α and β amylose, in the starch paste, he concluded that the number of swollen granules, high paste viscosity, and high α -amylose content of a starch paste are definitely interrelated.

Harrison (9) found in his viscosity measurements of wheat, corn, rice, and potato starch pastes that each showed a gradual increase in viscosity with time of heating up to a maximum point, after which there was a decrease. In the case of potato starch, there was a marked

⁴ CHAPMAN, O. W. Op. cit. (See footnote 3.)

decrease in viscosity which he explains as being caused by the great variation in size of the granules and the breaking up of the swollen granules as they flow down the capillary tube. When starch pastes were heated to a temperature just above their swelling point, the paste cooled, and the swollen granules separated by centrifuging, he found that the volume of the swollen granules was directly proportional to the viscosity of the paste, regardless of what starch was used.

There are many other factors which also affect the consistency of pastes, but this brief review of the work of previous investigators shows that the number and size of the swollen starch granules in a paste greatly influence the viscosity of that paste.

In order to measure the granule size of the starch in this study, a filar micrometer attachment for the microscope, which had been calibrated by the United States Bureau of Standards, was used. The length and width of a large number of raw starch granules were measured at a magnification of 537.5. The micrometer held a 20-millimeter ($\times 12.5$) eyepiece, and a 4-millimeter ($\times 43$) objective of the microscope was used. The results of these measurements are given in Table 1. The standard deviation in this table is the square root of the sum of the squared deviations from the average divided by the number of granules measured. The coefficient of variation is the standard deviation expressed as percentage of the average. The swollen granules of rice, dasheen, and corn were also measured with the 4-millimeter objective, but for wheat, potato and canna it was necessary to use the 16-millimeter ($\times 10$) objective, making the magnification 125 diameters. Table 1 also gives the measurements for the swollen starch granules.

TABLE 1.—Size of starch granules from various sources

RAW STARCH GRANULES								
Source of starch	Length				Width			
	Granules measured (N_i)	Average length (L_i)	Standard deviation (σ)	Coefficient of variation ($\frac{\sigma}{L_i}$)	Granules measured (N_i)	Average width (W_i)	Standard deviation (σ)	Coefficient of variation ($\frac{\sigma}{W_i}$)
	Number	Microns	Microns	Per cent	Number	Microns	Microns	Per cent
Potato.....	65	50.3	15.0	30.0	65	33.0	9.6	29.1
Canna.....	50	63.5	17.1	26.9	50	43.5	12.2	28.0
Corn.....	65	13.8	1.3	31.2	65	12.1	3.7	30.6
Wheat.....	50	13.5	7.5	55.6	50	11.2	6.3	56.3
Rice.....	50	5.7	0.89	15.6	50	4.8	0.81	16.9
Dasheen.....	50	4.1	1.0	24.4	50	3.3	0.93	28.2

SWOLLEN STARCH GRANULES								
Source of starch	Length				Width			
	Granules measured (N_i)	Average length (L_i)	Standard deviation (σ)	Coefficient of variation ($\frac{\sigma}{L_i}$)	Granules measured (N_i)	Average width (W_i)	Standard deviation (σ)	Coefficient of variation ($\frac{\sigma}{W_i}$)
	Number	Microns	Microns	Per cent	Number	Microns	Microns	Per cent
Potato.....	75	324.8	113.3	34.9	75	200.3	73.8	36.8
Canna.....	75	279.9	89.5	24.8	75	221.5	50.7	22.9
Corn.....	75	59.8	13.6	22.7	75	49.7	11.9	23.9
Wheat.....	75	70.0	28.6	40.9	75	56.9	23.6	41.5
Rice.....	55	10.5	2.3	21.9	55	8.8	1.8	20.5
Dasheen.....	80	9.3	1.7	18.3	60	7.5	1.3	17.3

The size of the raw granules of potato, canna, corn, wheat, and rice agree very well with that reported by Reichert (18). Barrett (2) states⁵ that the starch grains of taro varieties (including dasheen) range in size from 1 to 3 microns. Alsberg (1) in discussing the swelling of starch granules reports that potato swells about 40 times, canna 16.5 times, and wheat only 8 times its natural size. A comparison of the amount of swelling of the different starches used in this study was made by calculating the ratio of the swollen-granule size to the raw-granule size. These values for both the length and width of the granules are given in Table 2. From the results reported in Table 1 it is also evident that different starches swell to different degrees even though the size of their raw granules is somewhat the same. A study of the coefficient of variation in length and width of the swollen granules in Table 1 emphasizes the great variation in the size of starch granules in a given paste.

TABLE 2.—Ratio of size of swollen starch granules to size of raw granules in starches from different sources

Source of starch	$\frac{L_2^1}{L_1}$	$\frac{W_2^1}{W_1}$	Source of starch	$\frac{L_2^1}{L_1}$	$\frac{W_2^1}{W_1}$
Potato.....	6.46	6.07	Wheat.....	5.19	5.06
Canna.....	4.41	5.09	Rice.....	1.84	1.83
Corn.....	4.33	4.11	Dasheen.....	2.27	2.27

¹ Symbols refer to values in Table 1.

CONSISTENCY OF STARCH PASTES

Flow tests were made by Ruth E. Elmquist and the writer on the six starches—potato, canna, corn, wheat, rice, and dasheen—at one concentration (3.7 per cent). Pastes were made according to the method previously developed in connection with this study (17). In this procedure an accurately weighed sample of desiccated starch was mixed with a definite quantity of cold distilled water. This cold-water-starch suspension was poured slowly into a definite quantity of hot distilled water, kept in motion by a mechanical stirrer, and the paste was subsequently heated in a boiling-water bath for one-half hour. A buret consistometer similar to that of Herschel and Bulkley (12) was made by joining, with rubber tubing, a capillary tube to an ordinary 25-cubic centimeter buret. This was fitted into position in a long tube leading to the bottle which was to receive the starch paste coming through the consistometer. The entire apparatus was placed in a large glass-sided water jacket full of hot water kept in motion with a motor-driven mechanical stirrer. The temperature of the bath was maintained at 87° C., which was the temperature of sizing, and regulated to stay within $\pm 0.1^\circ$ of 87°. In order to prevent flow of the paste in the consistometer while it was coming to temperature in the bath, for the 15 minutes allowed, rubber tubing with its free end held tight by a pinch clamp was pulled over the top of the buret. To make a run, this rubber tubing was cut quickly and the descent of the meniscus observed. The time for the flow of the paste was taken at every 5 cubic centimeters for 25 cubic centimeters. Flow tests for corn,

⁵ This statement was based on original measurements made by B. J. Howard, Bureau of Chemistry and Soils, U. S. Department of Agriculture.

TABLE 3.—Flow-test data of starch pastes (3.7 per cent starch)

Starch	Density of starch pastes	Consistometer A					Consistometer B				
		Determinations averaged	Readings	Time of flow (t)	Average rate of flow (q)	Average pressure (P)	Determinations averaged	Readings	Time of flow (t)	Average rate of flow (q)	Average pressure (P)
	Gm. per c.c.	Number	C.c.	Seconds	C.c. per sec.	Gm. per sq. cm.	Number	C.c.	Seconds	C.c. per sec.	Gm. per sq. cm.
Potato ¹	.9804	4	0	0	.152	32.86					
			5	32.9	.118	27.64					
			10	75.3	.0910	22.41					
			15	137	.0472	17.17					
			20	243	.0342	11.91					
			25	450	.187	32.88					
Canna ¹	.9810	4	0	0	.141	27.65					
			5	26.7	.0984	22.42					
			10	62.2	.0868	17.18					
			15	113	.0868	17.18					
			20	201	.0287	11.92					
			25	375							
Corn	.9814	10	0	0	1.02	32.90		0	0	0.239	30.18
			5	4.9	.781	27.67		5	20.9	.174	25.04
			10	11.3	.538	22.44		10	49.6	.116	19.75
			15	20.6	.327	17.18		15	92.6	.0633	14.50
			20	35.0	.156	11.92		20	171	.0282	9.28
			25	67.0				25	362		
Wheat	.9807	5	0	0	1.52	32.87		0	0	.308	30.13
			5	3.3	1.28	27.64		5	13.6	.289	25.02
			10	7.2	.980	22.42		10	30.9	.209	19.73
			15	12.3	.641	17.17		15	54.8	.130	14.55
			20	20.1	.379	11.91		20	93.3	.0844	9.26
			25	33.3	.746	32.89		25	171	.172	30.15
Rice	.9812	4	0	0	.617	27.60		0	0	.129	25.03
			5	6.7	.455	22.43		5	29.0	.0924	10.74
			10	14.8	.299	17.18		10	67.0	.0588	14.56
			15	28.8	.166	11.92		15	122	.0282	9.26
			20	42.5				20	210		
			25	72.7				25	401		
Dasheen	.9813	7	0	0	1.06	32.89		0	0	.181	30.15
			5	4.7	.834	27.66		5	27.7	.139	25.03
			10	10.7	.640	22.43		10	63.8	.0906	19.74
			15	18.4	.424	17.18		15	114	.0625	14.66
			20	30.2	.250	11.92		20	194	.0314	9.26
			25	50.2				25	353		

¹ Flow tests for these starches were not made with Consistometer B.

wheat, rice, and dasheen pastes were made with two different capillaries because the pastes varied considerably in consistency. For potato and canna pastes only the larger capillary was used. Each determination was from a freshly prepared sample, and the results given in Table 3 are averages of from 3 to 10 determinations.

The consistometers were calibrated by flow tests with standard samples of oils supplied by the United States Bureau of Standards. Data on the calibrating liquids are contained in Table 4.

TABLE 4.—Calibrating liquids

Liquid	Temperature	Density (ρ)	Viscosity (η)	Flow time of 25 c. c. (t)	
				Consistometer A	Consistometer B
	° C.	Gm. per c. c.	Poise	Seconds	Seconds
Water.....	25	0.997	0.00894	6.1	25.7
Oil No. 1.....	25	.885	.560	119	769
Oil No. 2.....	25	.841	.187	41.5	268

The length of each capillary was measured with a millimeter rule. From the data obtained in the flow tests of the oils the diameters of the capillaries were calculated according to the method described by Herschel and Bulkley (12). The results are given in Table 5.

TABLE 5.—Dimensions of the capillaries used

Capillary for consistometer	A	B
Length in centimeters (l).....	5.92	3.27
Inside diameter in centimeters (d).....	.2020	.1133
Ratio $\frac{d}{l}$0341	.0348

The average head, h , for the two consistometers was also calculated by the equation of Herschel and Bulkley (12). The instrumental constant C and the kinetic energy constant K , the values for which are found in Table 6, were obtained from the data of the flow tests of oils given in Table 4 and from the equation $\frac{\rho}{\eta} = \frac{C}{K} \frac{1}{t - \frac{C}{K}}$, given by Clibbens

and Geake (4). In this equation, ρ represents the density, η the viscosity, and t the time of flow of the viscous material. The values of the constants C and K may be substituted in this equation in order to calculate the apparent viscosity of the starch pastes.

TABLE 6.—Constants for consistometers

Constants for consistometer	A	B
C (instrumental constant).....	183.6	1,188.0
K (kinetic energy constant).....	27.17	386.6

Since the viscosity of a plastic material is a variable depending on the relation between pressure and rate of flow, it was thought best to describe the consistency of each starch paste by its yield shear value, f , and its mobility μ . Herschel and Bulkley (11) working with rubber-benzene solutions and Bingham (3) with paints have shown that for some plastic materials these two terms are nearly constant in value and do not depend on the rate of flow or on the dimensions of the capillary used. It was hoped that this would also be true of starch pastes. The yield shear value and mobility for each starch paste was therefore calculated according to the following equations:

$$f = \frac{pdg}{4l} \quad \text{and} \quad \mu = \frac{128ql}{\pi g d^4 (P - p)}$$

where f = yield shear value (dynes per square centimeter)
 p = yield value (gram per square centimeter)
 g = acceleration of gravity = 981 centimeter per second per second
 d = diameter of capillary in centimeters
 l = length of capillary in centimeters
 q = rate of flow of paste (cubic centimeters per second)
 μ = mobility (square centimeters per dyne-second)
 P = average pressure of flow (grams per square centimeter)
 $P = p \times h$

When the rate of flow of the starch paste, q , was plotted against the average pressure, P , to obtain the flow-pressure diagram, the yield value, p , was obtained as the intercept of the upper straight portion of the graph, prolonged on the abscissa axis. Since the kinetic energy pressure correction for capillary A was only slightly greater than 1 per cent in the extreme case of wheat starch and for capillary B was considerably less, it has been ignored in these calculations. Figures 1 and 2 are flow-pressure diagrams for the starch pastes. Table 7 gives the calculated results of the consistency of the starch pastes. The values of the rate of flow, q , used in calculating the mobility, μ , in this table are taken from the flow-pressure diagrams rather than from the actual experimental values.

TABLE 7.—Consistency of starch pastes (3.7 per cent starch)

Starch	Consistometer A			Consistometer B		
	Yield value	Yield shear value	Mobility	Yield value	Yield shear value	Mobility
	(p)	(f)	(μ)	(p)	(f)	(μ)
	Gm. per sq. cm.	Dynes per sq. cm.	Sq. cm. per dyne-sec.	Gm. per sq. cm.	Dynes per sq. cm.	Sq. cm. per dyne-sec.
Potato.....	10.40	87.03	1.01			
Canna.....	10.60	88.70	1.25			
Corn.....	9.60	80.34	6.42	9.60	81.94	9.28
Wheat.....	6.05	50.63	8.61	6.05	51.64	12.37
Rice.....	6.80	56.90	4.25	6.80	58.04	5.88
Dasheen.....	6.15	51.47	5.82	6.15	52.49	5.97

Table 7 shows some differences in mobility and in yield shear value with the two capillaries (consistometers A and B). In mobility values alone, however, there was fairly close agreement with both capillaries for the rice and for the dasheen pastes. This may be accounted for in some measure by the fact that, as is shown in Table 1, these starch pastes are composed of smaller and more uniformly sized swollen granules than corn and wheat. The work of Herschel and Bergquist (10) agrees with the present study in so far as these authors also obtained varying results for rigidity and yield shear values on starch pastes when different capillaries were used. On the

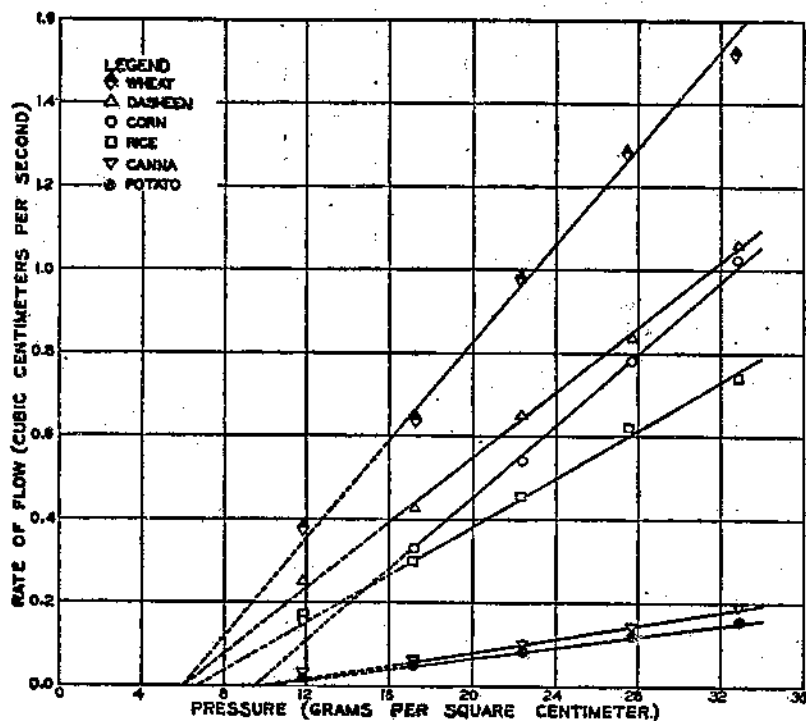


FIGURE 1.—Flow-pressure diagram of starch pastes (3.7 per cent starch), using consistometer A

other hand Farrow, Lowe, and Neale (6), under the special conditions of their experiment on flow tests of formaldehyde-treated starch pastes of various concentrations, obtained constant values with different capillaries. Their conditions such as kind of starch, concentration of paste, etc., are, however, quite different from those in the present study.

PENETRATING AND COATING POWERS OF STARCH PASTES

The comparative penetrating and comparative coating powers of the different starch pastes on fabric and on yarns reaveled from the fabric were determined qualitatively by microscopic examination of cross sections of the starched yarns and fabric. Other investigators (5, 8, 14, 24) have studied the penetrating and coating powers of

starch mixtures by this method, and some have determined the amount of penetration by quantitative methods.

Krais and Gensel (14) studied the sizing of cotton yarn using different starches and sizing mixtures. They reported that penetration of the size into the yarn is greater the higher the degree of dispersion of the starch in the size and the lower the viscosity of the sizing materials. Greater penetration was also observed with hydrolyzed

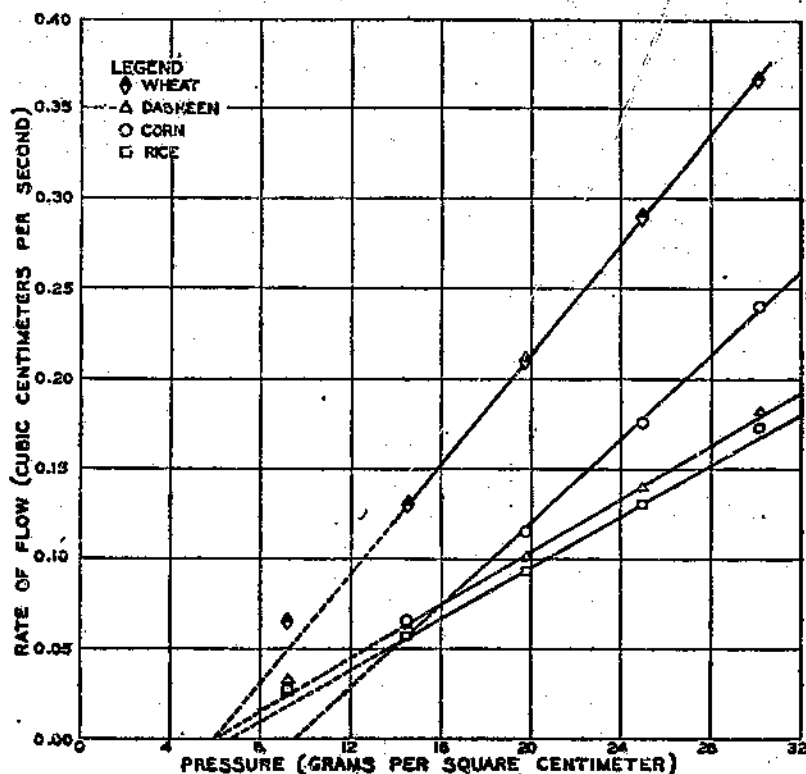


FIGURE 2.—Flow-pressure diagram of starch pastes (3.7 per cent starch), using consistometer B

than with unhydrolyzed starches and with a size containing wheat starch than with one containing potato starch.

Grimshaw (8) on sizing cotton yarns with various sizing mixtures found that yarns sized with corn starch were more deeply penetrated than most of those treated with potato starch.

Farrow and Jones (5) in an examination of the process of sizing cotton yarns on an experimental tape frame reported that the amount of sizing picked up by yarns depends on the construction of the yarn and on the viscosity of the size. They also stated that the penetration of the size into the yarns is determined by physical conditions which control flow, and is increased by pressure, by repetition of squeezing, or by prolonged pressure.

It is therefore generally understood that penetration of sizes into yarns and fabrics is influenced by the viscosity of the starch paste

and by such other factors as twist and construction of yarns, temperature of sizing, and concentration of the paste.

Parker (15) believes that penetration depends also on the size of the starch granule. He found that of raw-starch granules, rice starch penetrates cotton fabric most freely, and that the degree of penetration for wheat, maize, and potato starches decreases in the order given. Both the rice starch and the small granules of wheat starch penetrate the yarns of the fabric to a marked extent.

In this study the penetrating power of a starch is considered as the extent to which the starch paste has gone into the yarn or fabric and the completeness with which it has filled in the spaces between the fibers. By "coating" is meant the starch on a starched yarn that adheres to the outer portion of the yarn and surrounds it like a casing.

The fabric used for these penetration and coating studies was the same as that used in the stiffness studies. In the case of the yarns, both warp and filling yarns were raveled out from the fabric and the ends of each tied to glass stirring rods to keep them from twisting and tangling. They were then dipped in distilled water, dried, immersed in the starch paste where they were worked for one minute and left without manipulation for two more minutes. The excess paste was brushed off gently without applying pressure, and the yarns were dried under just enough tension to prevent tangling. The method followed in sizing fabrics for stiffness determinations was also used for the penetration measurements on the fabrics.

In preparing the yarns and fabric for cutting cross sections, it was necessary to mount them on cardboard frames in order to handle them better during the imbedding process. For this, frames were made as Kisser and Anderson (13) describe, and the yarns and fabric were placed for 12-hour periods in each of the following: 95 per cent alcohol, ether-alcohol solution, and ether-alcohol solutions of 2 per cent, 4 per cent, and 8 per cent celloidin. The samples on the frames were covered with 8 per cent celloidin in a stender dish and the celloidin allowed to thicken. The blocks of celloidin containing the samples were cut out, hardened in chloroform and double embedded in paraffin of 52° C. melting point. These double-embedded samples were then fastened to wooden blocks and kept in the cold until ready to use.

The sections were cut on a microtome to a thickness of 10 microns. They were spread and cemented to slides with the usual protein-glycerin mixture, and after drying were treated with xylol to remove the paraffin, and with ether-alcohol to remove the celloidin. The sections were stained with a dilute solution of iodine in potassium iodide and were mounted in glycerin jelly. In order to have permanent records of the sections, photomicrographs of 200 magnification were made. The photomicrographs had to be taken immediately after staining because the iodine stain fades quickly.

The warp yarns in the fabric studies were more tightly twisted and therefore more uniform in size and shape than the filling yarns. Since the construction of a yarn is a factor influencing the way starch pastes penetrate and coat yarns, it seemed that the warp yarns showed the penetration and coating more fairly than the filling yarns. Plate 2, B to G, inclusive, shows photomicrographs of cross sections of warp yarns sized with their respective starches. The dark area shows the starch stained with iodine. Although the lumens of the fibers appear dark, it is due to shadows and not to stained starch. This is evident

when one compares the pictures of starched yarns with Plate 2, A, which is a cross section of an unsized warp yarn. Plate 3, A to G, inclusive, shows the cross section of the unsized and sized filling yarns.

From direct observation with the microscope, the penetrating powers of the starches seem to follow the order of dasheen, rice, wheat, corn, canna, and potato, with dasheen starch showing greatest penetration and potato the least. Canna and wheat starches have practically equal coating powers, followed in order by corn, rice, and then dasheen. Evidently potato starch does not adhere well to cotton yarns because the yarns starched with potato did not show a coating.

Conclusions drawn concerning the order of the degree of penetrating and coating powers of the starches must be based on critical observation of the sized yarns and fabric under the microscope. It is almost impossible to make any classification from photomicrographs alone because of the difficulty of distinguishing between the shadows and the small amounts of starch penetrating the yarns. This is particularly noticeable for a starch like rice which is distributed evenly through the entire yarn. But for wheat starch, which has very uneven distribution between the fibers, photographic reproduction brings out a sharp contrast due to the well-defined dark areas. The fact that some starches show very similar penetrating and coating powers also adds to the difficulties of obtaining characteristic photomicrographs.

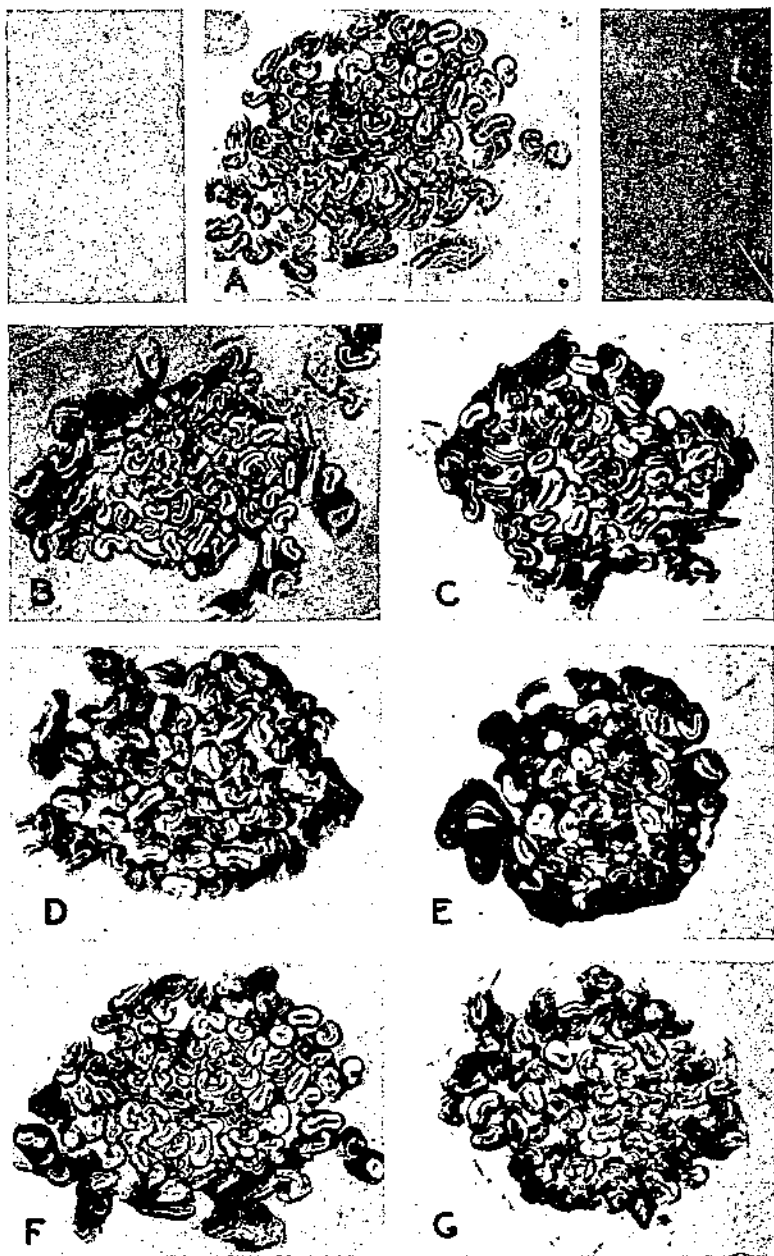
The order of the penetrating and coating powers of the starches appeared on critical examination with the microscope to be the same for the fabric as for the yarns. It therefore seemed unnecessary to include photomicrographs of the sized fabric in this bulletin.

STIFFNESS OF SIZED FABRICS

Stiffness measurements of sized fabrics were made according to the method previously developed in this division (17). Starch pastes of 3.7 per cent concentration were prepared, and desized fabric of the same construction as that used in the former study was sized with the starch pastes. The sample of cloth to be tested was first dipped into cold distilled water, wrung through a carefully adjusted wringer, immersed in the starch paste for three minutes, put through the wringer again, and the sized fabric then stretched on a wooden frame to dry. The most constant results were obtained when the fabric was slowly stirred during the first of the three minutes it was in the starch paste. Farrow and Jones (5) found in their study on sizing of cotton yarns that penetration of the size is greatly affected by the pressure and speed of the sizing machine. Therefore, great care was taken in these tests to keep the pressure and speed of the rolls constant throughout the whole series of experiments. These sized fabrics were cut into strips 2 inches wide and 8 inches long. They were then hung overnight and tested in a humidity room⁶ maintained at 50 per cent relative humidity and at a temperature of 70° C.

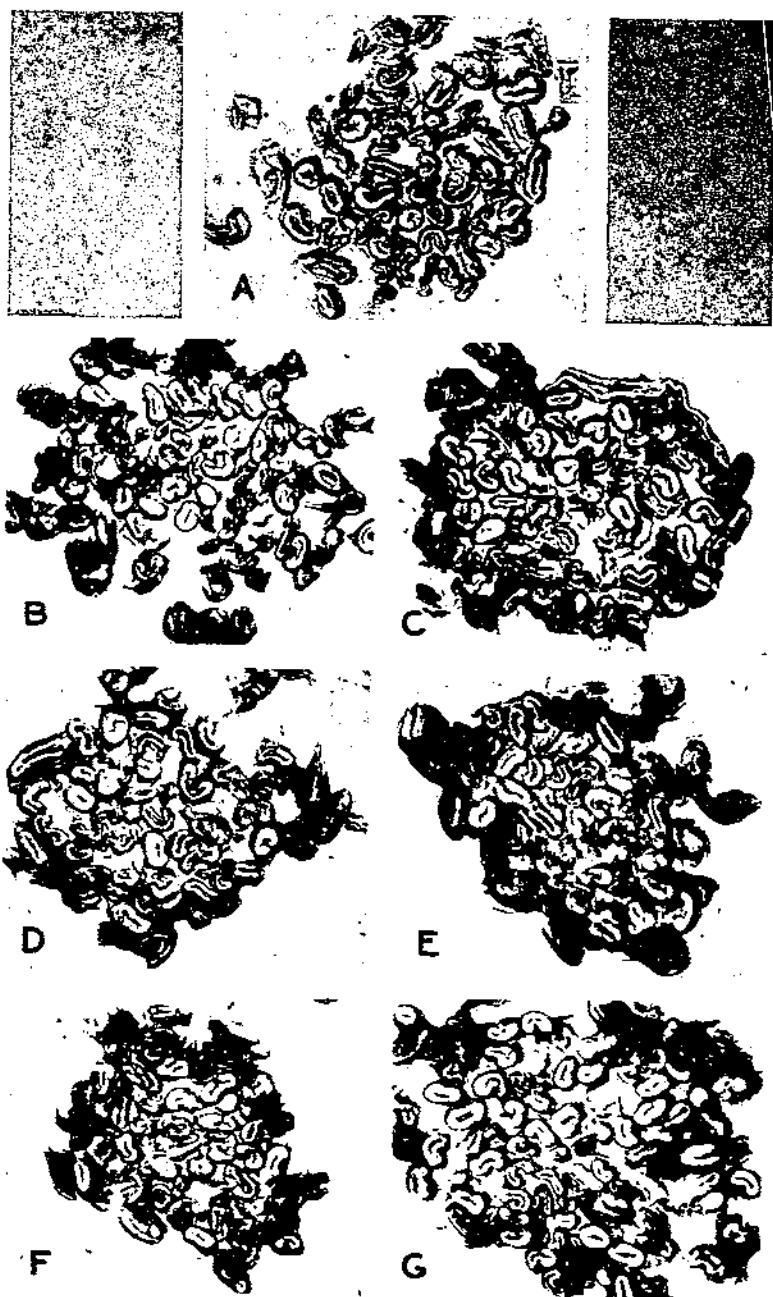
Stiffness was determined on a stiffness-testing apparatus devised by Peterson and Dantzig (17). The working principle of the tester depends on the deformation at an angle of 45° of a horizontally supported strip of material bending under its own weight. The apparatus consists of a vertical board provided with a metal shelf 2½

⁶ The constant temperature and relative humidity room, in the Division of Tests and Technical Control of the Government Printing Office.



PHOTOMICROGRAPHS OF CROSS SECTIONS OF WARP YARNS SHOWING PENETRATING AND COATING POWERS OF STARCHES

A, Warp yarn unsized; B to G, warp yarn sized with potato starch (B); canna starch (C); corn starch (D); wheat starch (E); rice starch (F); and dasheen starch (G). All $\times 200$.



PHOTOMICROGRAPHS OF CROSS SECTIONS OF FILLING YARNS SHOWING PENETRATING AND COATING POWERS OF STARCHES

A, Filling yarn unsized; B to G, filling yarn sized with potato starch (B); canna starch (C); corn starch (D); wheat starch (E); rice starch (F); and dasheen starch (G). All $\times 200$.

inches wide extending from the upper corner at an inclination of 45° to the horizontal. The fabric to be tested is slowly fed through a clamp consisting of two rubber rolls mounted at the upper end of the metal shelf. In making this test, the definite position of the cloth at that point where the free end of the strip barely touches the metal shelf must be noted. Since it has been shown mathematically that projected strips of starched fabrics follow elastic laws and that their stiffness is proportional to the cube root of the elastic modulus and to the radius vector, a scale bearing these relations was made and marked on the apparatus. In this way, stiffness can be read off directly and is expressed by a value equal to 0.43 times the millimeter length of the projected fabric supporting its own weight at a 45° angle.

Peirce (16) has recently described an instrument, called a flexometer, which is similar in principle to the stiffness tester. This instrument makes a direct measurement of the angle through which a sample of cloth droops when a definite length is held out over an edge. The bending length and the flexural rigidity or stiffness of the fabrics tested can be obtained by simple calculation from the formulas which he has developed.

Table 8 shows the stiffness results obtained with fabrics starched with rice, wheat, corn, and potato starch as compared with those values reported previously. When the fabric was worked slowly during the first of the three minutes it was in the size, the stiffness value was slightly lower than when the fabric was worked in the size for the entire three minutes. The stiffness values for canna and dasheen starch are reported in this table for the first time.

TABLE 8.—Stiffness produced in a fabric by starch pastes

Starch	Determined by Peterson and Dantsig (17)				Determined by author							
	Fabric in paste three minutes with some stirring				Fabric in paste three minutes with stirring for one minute				Fabrics stirred in paste three minutes			
	Samples (N_1)	Mean stiffness of N_1 samples (M_1)	Standard deviation (σ_1)	Coefficient of variation ($\frac{\sigma_1}{M_1}$)	Samples (N_2)	Mean stiffness of N_2 samples (M_2)	Standard deviation (σ_2)	Coefficient of variation ($\frac{\sigma_2}{M_2}$)	Samples (N_3)	Mean stiffness of N_3 samples (M_3)	Standard deviation (σ_3)	Coefficient of variation ($\frac{\sigma_3}{M_3}$)
	Number			Per cent	Number			Per cent	Number			Per cent
Potato.....	160	36.7	4.5	12.1	27	37.5	1.7	4.3	39	43.5	1.3	3.8
Canna.....					34	48.7	2.2	4.5	49	49.9	2.0	4.0
Corn.....	76	42.7	2.0	4.6	13	40.9	0.8	2.0	64	42.0	1.2	2.9
Wheat.....	160	42.8	2.7	6.3	15	41.7	0.9	2.2	35	42.5	1.1	2.6
Rice.....	80	42.9	2.1	4.8	15	41.1	1.0	2.4	56	42.1	1.0	2.4
Dasheen.....					32	47.1	1.8	3.8	28	49.3	1.5	3.0

* Mean stiffness of N samples expressed as $0.430 \times$ millimeter length of projected fabric supporting own weight at 45° angle.

DISCUSSION

A comparison is given in Table 9 of the stiffness of the fabric at one starch concentration with the penetrating and coating powers of the starches, the size of the swollen granules, and the consistency of the starch pastes.

TABLE 9.—Comparison of fabric stiffness with properties of starch pastes

Starch	Mean stiffness produced in a fabric expressed as 0.43×millimeter length of projected fabric supporting own weight at 45° angle (M)	Length of swollen starch granules						Penetrating power of pastes into yarns and fabric (6=greatest penetration)	Coating power of pastes on yarns and fabric (4=greatest coating)	Paste consistency (consistometer A)		
		Granules measured (N ₂)	Average length (L ₂)	Standard deviation (σ)	Coefficient of variation $\left(\frac{\sigma}{L_2}\right)$	Average length minus standard deviation (L ₂ -σ)	Average length plus standard deviation (L ₂ +σ)			Yield value (p)	Yield shear value (q)	Mobility (s)
		Number	Microns	Microns	Per cent	Microns	Microns			Gm. per sq. cm.	Dynes per sq. cm.	Sq. cm. per dyna-sec.
Potato.....	37.5	75	324.8	113.3	34.9	211.5	438.1	1	1	10.40	87.33	1.01
Canna.....	48.7	75	279.9	69.5	24.8	210.4	349.4	2	4	10.60	88.70	1.25
Corn.....	40.9	75	59.8	13.6	22.7	46.2	73.4	3	3	9.00	80.34	6.42
Wheat.....	41.7	75	70.0	28.6	40.9	41.4	98.6	4	4	6.05	50.63	8.61
Rice.....	41.1	55	10.5	2.3	21.9	8.2	12.8	5	2	6.80	56.90	4.25
Dasheen.....	47.1	60	9.3	1.7	18.3	7.6	11.0	6	1	6.15	51.47	5.82

In considering first the size of the swollen granules and the penetration of the pastes, it will be noted that the order of size for the average length minus the standard deviation, $L_2 - \sigma$, of the swollen granules, is the same as for the penetration of the starch pastes in the yarns and fabric. It seems, therefore, that at this concentration of starch paste, there is a definite relation between the size of the swollen granules and the penetration of the starch paste into the yarn. The swollen granules of dasheen paste are the smallest and the paste appears to penetrate most thoroughly, whereas the potato paste (largest granules) scarcely penetrates at all.

The relation between the size of swollen granules and the coating of the starches on the yarns is also shown by the fact that the average length plus the standard deviation, $L_2 + \sigma$, of the swollen granules has the same order as the coating power of the starches. Since penetration and coating are almost opposite terms, it might seem that a starch paste which penetrates would not necessarily coat the yarn at the same concentration and temperature. That would be the case if the starches were composed of uniformly sized granules, but, as is shown by the coefficient of variation in length and width of the swollen grains in Table 1, the granules of some starches have a great range in size. In the case of wheat starch this coefficient of variation in length of the swollen granules is very high (40.9 per cent) which makes the average length show a range from 41.4 to 98.6 microns. The fact that wheat paste is composed of very small granules as well as large ones doubtless accounts in part for its consistency, and its penetrating and coating powers.

Although the swollen granule of canna starch is very large in size it seems to coat about the same as wheat. Potato starch, which penetrates the least of any of these starches, scarcely coats at all. Probably this is due to the fact that the swollen potato granules are very large. They are too large to penetrate, and they slough or dust off instead of adhering to the yarn. This accounts for the low stiffness in the fabric starched with potato starch. Doubtless if the potato starch would adhere and coat the yarns, the stiffness of the fabric would be greater than that obtained with canna starch.

In a general way the mobility of the paste and the coating of the yarns follow the same order, whereas yield shear value and penetration are about in the same order.

Fabrics seem to be stiffened both by starches having penetrating powers and by those having coating powers. Dasheen, which penetrates most thoroughly and coats the least of the starches studied, and canna, which has little penetrating power but coats very well, have stiffness values very nearly the same. In the case of dasheen the stiffness is due to the penetration of the starch, and in the case of canna to the coating power.

These facts are undoubtedly of importance both in the finishing of cotton fabrics in the mill and in the refinishing of them in the laundry. The selection of a starch to be used depends to a large extent on the information available as to the degree of its coating and its penetrating powers. Whether or not a starch which penetrates well or one which has high coating power is chosen depends on the type and construction of the fabric to be sized. It is hoped that the experimental data given in this bulletin will show the need for scientific information by which starches may be selected.

SUMMARY

Some physical properties of six starches and their pastes have been studied. Length and width measurements of both the raw and swollen starch granules were taken by means of a filar micrometer attachment to the microscope, consistency determinations of the starch pastes at one concentration and temperature were made by measuring their rate of flow through capillaries, and the penetrating and coating powers of the starches were determined by examining cross sections of the sized yarns and fabric. The relation between these properties of starch pastes and the stiffness in fabrics produced by the pastes was observed.

The study shows that the stiffness of a sized fabric depends on the penetrating and coating powers of starch pastes, and these factors in turn depend in a general way on the consistency of the pastes. The findings indicate also that the size of the swollen starch granules bears a definite relation to the penetrating and coating powers of the starch pastes. Further evidence is given that the consistency of starch pastes depends on the size of the swollen starch granules.

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END