RAYON[1] AS A PAPER-MAKING MATERIAL

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ABSTRACT

Laboratory tests were conducted at the bureau to determine the paper-making quality of rayon when treated the same as rags are in the production of fine papers. Cotton and linen fibers are commonly used for papers, such as bonds and ledgers, on account of their strength and durability. In addition to their value in these respects, they are used in the manufacture of papeteries and other fine writing papers also to impart softness and other desirable characteristics of surface and finish. The presence in rag stock of any fiber which would adversely affect these qualities of the paper is undesirable. Since increasing amounts of rayon are being found in the rags and textile waste used in rag-paper manufacture, the test data are believed to be of value to that industry.

Samples representative of the four general processes of rayon manufacture—viscose, nitrocellulose, cuprammonium, and cellulose acetate—were included in the tests. The equipment employed in the preparation of the pulp and its subsequent conversion into paper was on a laboratory scale, and is that used at the bureau for the preliminary tests of all paper-making materials under investigation. The caustic soda and lime process were each used in the cooking operation. Paper-making stock was prepared from the pulp, alone and in admixture with sulphite pulp, and converted into paper on a sheet mold.

Owing to loss of strength when wetted the rayon filaments tended to break into short lengths during the preparation of the paper-making stock without the fibrillation and fraying necessary for good felting properties. As a consequence the all-rayon sheets lacked the strength to withstand the handling necessary in the pressing and drying operations and the pliability characteristic of rag papers. Likewise, sheets made of rayon in admixture with sulphite pulp were considerably weaker than those made from sulphite alone.

The test data indicate that rayon is valueless in the rag stock for high-grade papers and may actually be detrimental to their quality.

CONTENTS

I. INTRODUCTION

The increasing amounts of rayon intermixed with other textile fibers in the rag stock used in paper making has become a matter of

[1] The term "rayon" as used in this paper conforms to the definition by the American Society for Testing Materials, Tentative Standards 1929, p. 721.

some concern to rag-paper manufacturers. Cotton and linen fibers are commonly used for papers, such as bonds and ledgers, on account of their strength and durability. In addition to their value in these respects they are used in the manufacture of papeteries and other fine writing papers also to impart softness and other desirable characteristics of surface and finish. The presence in rag stock of any fiber which would adversely affect these qualities of the paper is undesirable. Rayon plays an important part in textile fabrics. The detection of it in the sorting of rags, however, especially used rags, is difficult. As a consequence the rags and textile waste used by the rag-paper industry may frequently contain a considerable quantity of rayon fiber. Information as to its paper-making quality when it is subjected to the same treatment as that given the cotton and linen in the paper manufacturing processes is therefore of interest to rag-paper manufacturers. In response to requests for such information, tests to obtain the necessary data have recently been made at the bureau. The following article describes the tests and includes also a brief outline of the manufacture and use of rayon.

II. PRODUCTION AND GENERAL CHARACTERISTICS OF RAYON

1. PRODUCTION AND CONSUMPTION

Production statistics of commercial textile fibers indicate that rayon is now established as a leading raw material in the textile industry. It is included with silk, wool, cotton, and flax as one of the five most important textile fibers of the world.[2] Alone, or in combination with other fibers, it is employed for almost every purpose for which a textile fabric is utilized.

Practically the entire textile industry, with the exception of the manufacture of certain woolen goods, is consuming rayon at the present time. Used in combination with other fibers it is said to be of advantage either in enhancing the value of the fabric produced or in reducing the cost of the product. Of particular interest to the paper manufacturer is the fact that an increasing number of cotton mills are using rayon in large quantities, and that sales of cotton-rayon mixtures are steadily increasing.[3] Fabrics reported to contain appreciable quantities of rayon—used independently of other fibers or in a supplementary way—and which the rag-paper manufacturer uses, include underwear, hosiery, shirtings, dress piece goods, linings, curtains, bedspreads, blankets, and draperies.

2. PROCESSES OF MANUFACTURE

Textile fibers are of two general classes—plant and animal. Cotton and linen have been the chief contributors of plant origin for articles of clothing and household furnishings, while wool and silk have led all animal fibers. Rayon is a synthetic fiber. The basic

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[2] The production of rayon in the United States during 1928 was 97,701,250 pounds, comprising 28 per cent of the world's total—347,940,000 pounds. Information on the actual sales of the various manufacturers indicates that this quantity was not only made but was consumed. Textile World, **75**, pp. 734-736; Feb. 2, 1929.

[3] A recent article published by the Metropolitan Life Insurance Co. states that the proportion of American rayon production going to the cotton goods trade was 22 per cent in 1927. Rayon: A New Influence in the Textile Industries, p. 17.

raw material used in its manufacture is either cotton or wood pulp, the fundamental constituent of which, as of all plant fibers, is cellulose. The cotton cellulose used is chiefly in the form of linters, the short fibers adhering to the cotton seeds after ginning; the wood pulp best adapted is principally that obtained from spruce by the sulphite process.[4] It is estimated that between 80 and 90 per cent of the world's production of rayon is from a wood pulp base.[5]

The cellulose base is treated chemically to produce a viscous substance, which is forced through minute apertures to form filaments. The filaments are hardened either by evaporation of the solvent or by some kind of precipating bath, and then twisted together into a continuous thread.

From a chemical point of view the rayon fibers are classified into two groups—those which are made from the cellulose in a modified physical form and those for which the raw material is converted into a different chemical product, a cellulose ester, before being made into the filaments. The groups differ in the solvents and methods used in converting the cellulose into plastic form rather than in the manner of forming the filaments. The first group includes the rayons manufactured by the processes designated as viscose, nitrocellulose, and cuprammonium, and the latter is represented by the cellulose-acetate product. The first three types of rayon are somewhat similar to each other in physical and chemical properties, but cellulose-acetate rayon has entirely different characteristics. The following brief description of the four methods of making rayon indicates further the differences in the resultant commercial types.

(*a*) *Viscose.*—The viscose process is the only one in which a wood-pulp base is commonly employed. It is the most widely used of the processes of manufacture. The wood pulp is first treated with a strong solution of caustic soda. After the removal of the excess alkali, the product, in the form of alkali cellulose, is ground into a crumblike mass, aged, and subsequently mixed with carbon bisulphide to form cellulose xanthate. The resultant gelatinous mass is dissolved in a dilute caustic-soda solution to form the spinning substance. The filaments emerging from the orifices do not naturally coagulate and are, therefore, passed through a bath for this purpose. The bath usually consists of sulphuric acid, some suitable salt, such as sodium sulphate, and glucose. The sulphur is removed from the filaments in a bath of sodium sulphite.

(*b*) *Nitrocellulose.*—In the nitrocellulose process cotton linters are nitrated by a mixture of nitric and sulphuric acids to form nitrocellulose. After a thorough washing this product is then dissolved in an alcohol-ether mixture to form a viscous liquid which is the spinning substance. A current of warm air causes the ether and alcohol content to evaporate from the filaments. Since the filaments are extremely flammable the nitrate element is next removed. Sodium hydrosulphite is the chemical most extensively used for the denitration.

(*c*) *Cuprammonium.*—The solvent for the cuprammonium process consists of an ammoniacal copper-oxide solution. After the cotton has been purified by a caustic-soda solution and bleached it is mixed

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[4] Sulphite is a term applied to a chemical pulping process whereby wood is reduced to pulp for paper-making by heating under pressure with a solution of acid sulphite of calcium, or magnesium.
[5] Rayon: A New Influence in the Textile Industry; p. 8, Metropolitan Life Insurance Co.

with the solvent to form the spinning material. The filaments are passed through a sulphuric acid bath for hardening.

(*d*) *Cellulose acetate*.—In the cellulose acetate process the raw material—cotton, wood pulp, or rayon waste—is mixed with acetic acid, acetic anhydride, and a catalytic agent. The resulting cellulose acetate is then mixed with acetone to form the spinning solution and after being forced through the apertures is coagulated into filaments by a current of warm air.

III. LABORATORY PAPER-MAKING TESTS

1. FIBROUS MATERIALS

The fibrous materials employed in the paper-making tests consisted of rayon and sulphite pulp, mixed together in different proportions, and in one case, for comparative purposes, a soda-sulphite mixture. The rayon included samples of spinning waste, in the form of thread, from the viscose, nitrocellulose, and cuprammonium processes, and cloth waste from the acetate process. The rayon was supplied by commercial companies, whose assistance is hereby gratefully acknowledged.

2. EQUIPMENT

The usual preliminary treatment given the rags in the manufacture of high-grade rag paper consists of sorting into grades, cutting to suitable size, dusting to remove dirt, boiling to remove nonfibrous constituents and soften the fiber, washing to eliminate the noncellulose impurities contained in the boiling solution, bleaching to remove residual impurities and whiten the fibers, and beating to separate the fibers and reduce them to the length and condition required for satisfactory felting in the forming of the paper. The beaten pulp is then diluted to the proper consistency and the fibers are deposited on wire cloth and thereby converted into paper on a Fourdrinier paper-making machine if the work is on a commercial scale; on a sheet mold if sheets are desired for laboratory experimental study only. After being pressed and dried the paper is then ready for use.

In the experimental tests described in this article the rayon was treated the same as rags are in making fine papers. The equipment employed in the preparation of the pulp and its subsequent conversion into paper was on a laboratory scale and consisted of a small rotary boiler, one-half pound beater (capacity one-half pound of dry fiber), fiber-sheet mold, sheet press, and dryer. This equipment is used at the bureau for the laboratory paper-making tests of all materials under investigation and except for differences noted herein is illustrated and fully described in Bureau of Standards Technologic Paper No. 340, Caroá Fiber as a Paper-Making Material, pages 326 to 334.

3. PROCEDURE

(*a*) *Cooking*.—The cooking process, which dissolves the noncellulose material and softens the paper-making fiber, was also similar to that described in Technologic Paper No. 340, and was representative of paper-making practice in the production of rag papers. Solutions of caustic soda (sodium hydroxide) and of lime (calcium oxide) were used to effect the chemical cleansing. The amounts of caustic soda and lime employed were 2 per cent and 6 per cent, respectively, based on the weight of fiber in the furnish. The constant factors for each cook, whether caustic soda or lime was employed, were as follows:

Weight of bone-dry material	pound	1
Volume of water	liters	6
Cooking temperature	°F	250–260
Cooking pressure	pounds	30
Time for raising to cooking temperature	hour	1
Duration of cooking temperature	hours	4
Time for cooling	hour	½

In preparing the lime solution a small amount of the water was used for slaking, and the remainder was later added to and thoroughly mixed with the slaked lime. The mixture was screened to remove objectionable foreign particles as it was charged into the boiler. For the caustic cooks the caustic soda was dissolved in the water and the solution was likewise screened as it was poured into the boiler.

The cooked pulp was washed and samples were taken for moisture determination. From the weight of the wet material and the moisture value the fiber loss was determined.

(*b*) *Bleaching.*—The pulp was bleached with from ½ to 1 per cent of bleaching powder, containing 35 per cent of available chlorine.

(*c*) *Beating.*—The different pulps used to obtain the fiber composition desired were blended in the beater. The beating consisted of only brushing out the fibers until there were no lumps or knots and the stock was in condition to produce a well-formed sheet. A beater furnish consisted of pulp equivalent to 135 g weight bone-dry, mixed with water to 1.75 per cent concentration.

There is a marked difference in the strength of dry and wet rayon. For example, the fibers lose from 45 to 75 per cent of their breaking strength when wet, although they regain their original strength upon becoming dry.[6] Because of this characteristic no attempt was made to develop the maximum strength of the wood fibers, as doing so would have destroyed the strength of the rayon pulp.

(*d*) *Molding, pressing, and drying test sheets.*—The method employed in converting the paper-making stock into test sheets differed from that described in the before-mentioned publication only in the procedure used in removing and pressing the sheet formed on the mold. The sheet of pulp on the mold was covered with a napless felt and upon it a perforated metal plate, a couching frame, was placed. The couching frame, felt, sheet of pulp, and wire cloth were then removed as a unit, inverted, and replaced on the mold base. Suction was again applied and the fiber sheet was transferred to the felt thereby.

The fiber sheet and felt were removed together and placed felt down on a metal plate. Another felt was laid over the sheet of pulp and upon it another metal plate. This process was repeated until there were five sheets of pulp in the stack. The stack of plates, sheets, and felts was placed in a hydraulic press and subjected for one minute to a pressure of 200 lbs./in.^{2} The paper sheets were then removed and dried.

[6] Moiŝ H. Avram, The Rayon Industry, p. 95. Published by D. Van Nostrand Co., 1927.

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4. TEST RESULTS

(a) *Fiber loss.*—The loss of rayon fiber in the cooking and washing operation was from 4 to 12 per cent for all the material tested. For tests conducted on a commercial scale the loss would doubtless be much greater. The rayon filaments would be broken into shorter lengths and the resultant fine material carried off with the wash water.

(b) *Tabular data on paper made.*—Data on the finished papers are given in Table 1 and include all the commonly applied paper tests. The methods employed are the standard test procedures used in the testing of paper. The strength measurements were made on air-conditioned test specimens at 65 per cent relative humidity and 70° F. temperature.

TABLE 1.—*Test data on hand-made sheets*

Test No.	Rayon sample	Chemical used in cooking	Fiber composition		Physical tests on finished paper											
			Sulphite	Rayon	Weight (25 by 40 inches, 500 sheets)	Bursting strength	Ratio of bursting strength to weight (25 by 40 inches, 500 sheets)	Thickness	Folding endurance[1]		Tensile strength[1] (strip 15 by 100 mm)		Elongation[1] (strip 15 by 100 mm)		Tearing strength[1]	
									Long direction	Short direction	Long direction	Short direction	Long direction	Short direction	Long direction	Short direction
			Per cent	*Per cent*	*Pounds*	*Points*	*Per cent*	*Inch*	*Double folds*	*Double folds*	*kg*	*kg*	*Per cent*	*Per cent*	*g*	*g*
1	Viscose	None	70	30	62.5	26.3	42.0	0.0065	35	50	4.12	3.95	3.4	3.4	71.2	70.0
2	do	Caustic soda	70	30	65.2	24.9	38.2	.0071	18	23	3.74	3.71	2.9	3.0	70.6	72.0
3	do	Lime	70	30	64.6	25.9	40.0	.0070	21	23	3.91	3.92	3.1	3.1	65.2	63.6
4	Cuprammonium	Caustic soda	70	30	66.3	28.0	42.3	.0071	28	24	3.96	3.85	3.5	3.2	74.8	72.0
5	do	Lime	70	30	64.1	24.2	37.7	.0070	20	21	3.59	3.84	2.9	3.0	61.6	66.0
6	do	do	30	70	60.6	8.9	14.7	.0084	1	1	1.49	1.56	1.6	1.8	36.6	36.6
7	Nitrocellulose	Caustic soda	70	30	60.5	22.0	36.4	.0071	22	19	3.40	3.50	3.0	3.0	65.2	68.4
8	do	Lime	70	30	63.8	23.4	36.7	.0076	17	17	3.76	3.81	3.2	3.3	70.8	73.6
9	Cellulose acetate	Caustic soda	70	30	58.9	21.3	36.2	.0060	11	14	3.70	3.60	3.1	2.9	36.8	36.6
10	do	Lime	70	30	60.2	24.7	41.0	.0054	21	28	4.40	4.10	3.2	3.2	36.2	39.5
11	None (sulphite)	None	100	----------	61.4	35.3	57.5	.0056	90	160	4.95	4.63	2.9	3.3	70.4	67.6
12	None (sulphite-soda)	do	70	{None. Soda,30}	62.8	26.8	42.7	.0061	35	35	4.13	4.16	3.4	3.1	66.8	66.0

[1] The average values in the long and short directions (which should be equal in the ideal case) are given separately merely to indicate the uniformity of the sheet in different directions.

An attempt was made to mold a sheet from all-rayon stock, but owing to the low strength of the wet pulp the sheet did not withstand the handling necessary in the remaining steps of the conversion process. Even when dry the paper broke readily when handled. For test sheets, therefore, the rayon was used only in admixture with a stronger pulp, sulphite. Sheets were also made from 100 per cent sulphite pulp and from a mixture of sulphite and soda pulps, 70:30, for comparative purposes. As is apparent from Table 1 and Figure 1 the addition of rayon weakened the sheet very appreciably and when the amount was increased to 70 per cent, test No. 6, the paper had very little strength. The lower strength of the sheets containing rayon was most marked in the folding endurance and the resistance to bursting, the tests most commonly applied to evaluate the quality of paper.

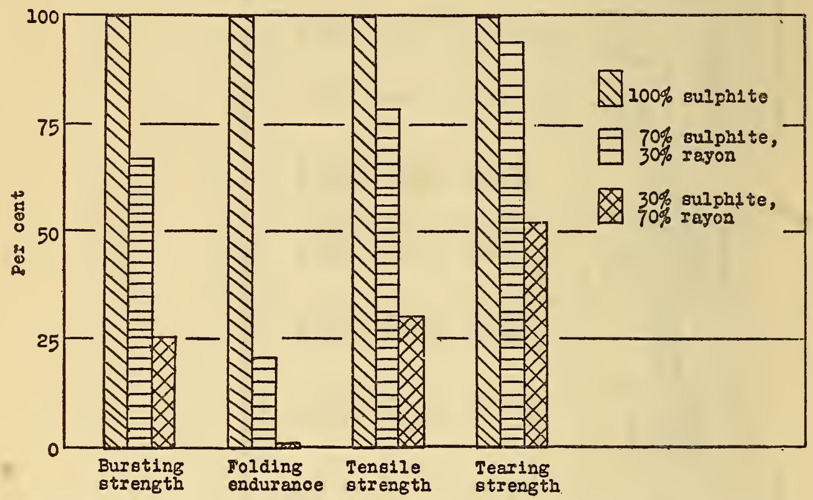

FIGURE 1.—*Comparative strengths of sulphite and sulphite-rayon handmade paper*

For each test the strength of sulphite sheet is taken as 100.

IV. DISCUSSION

The test data indicate that rayon is not suitable for use as paper-making fiber. The strength of the finished paper depends upon the length, strength, and flexibility of the fiber from which it is made, and the condition of the fiber ends. The more brushed out or frayed the ends, the better the contact between adjacent fibers when interlaced in the formation of the sheet. Owing to the characteristic loss of strength of rayon when wet the filaments tend to break into short lengths during the preparation of the paper-making stock, without the fibrillation or splitting of the fibers and the fraying of the ends necessary for good felting properties. As a consequence the molded fiber sheet lacks the strength necessary for the handling in the remaining operations of the paper-making process. This deficiency would be even more marked if the stock was run over a paper machine, because of the increased strain to which the sheet would be subjected.

If rayon is used in admixture with other rag fibers and submitted to the paper-making treatment usually given such materials, excessive reduction in length of fiber results, and, consequently, considerable fiber is lost in the pulping operations. Also, some of the short fragments remaining in the stock may stand on end in the formation of the sheet and appear as fuzz on the finished paper.

Pliability is an essential property of rag paper. Rayon treated as rags are, and in making fine papers, lacks the flexibility characteristic of cotton and linen fibers, and may, therefore, be not only valueless in rag stock, but actually detrimental to the quality of the paper.[7]

WASHINGTON, September 18, 1929.

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[7] Although rayon in rayon-cotton mixtures is apparently a complete loss to the paper manufacturer, all-rayon rags or fibrous waste intermixed with paper-making materials could doubtless be profitably disposed of if separated from the cotton and linen in the sorting process. Rayon waste is established as a commodity in the textile industry and the uses to which it is put are many. Converted into yarn by the methods used for discontinuous fibers and spun with wool waste it is widely employed in mackinaw coats, sweaters, boys' outer garments, blankets, carpets, and plushes. Some of the woolen goods in which cotton was once used now use no cotton whatsoever, but contain rayon instead. A real demand for rayon waste exists and has proved greater than the supply available.