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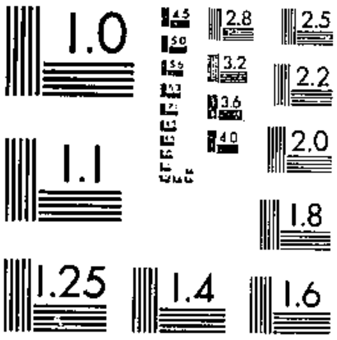

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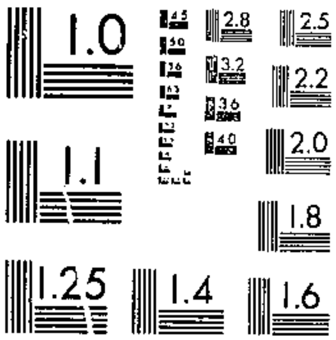

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Commercial Cotton-Bleaching Processes and Their Effect on Fabrics[1]

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CONTENTS

INTRODUCTION

At no period in the history of the textile industry has there been greater demand than at present for information regarding the physical and chemical changes in fabrics during the manufacturing and finishing operations. The characteristics of acceptable fabrics, measures for detecting poor lots of goods, the effects of certain operations or processes, and causes of abnormalties, are some of the specific problems involved.

In the alterations, revisions, and improvements of textile processes so far accomplished, comparatively little account has been taken of the changes that occur in the fabrics. The normal control tests capable of detecting unsatisfactory fabrics are frequently not sensitive enough to detect the slight variations which indicate the starting point of degradation. Only the highly sensitive and highly accurate techniques of the research laboratory are capable of detecting this initial deterioration. If, as is believed, degradation starts in the bleachery, application of research analyses to fabrics as they progress through the

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[2] Appreciation is expressed to Charles F. Goldthwait, for making arrangements for the commercial processing of the fabrics; to the Colloidal Properties Section of the Cotton Fiber Research Division of the Southern Regional Research Laboratory for cellulose and fluidity analyses; to Dorothy Legendre for assistance in breaking strength and reflectance measurements; and to Kathryn L. Baringer for assistance in preparing the manuscript.

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bleachery should result in a more detailed knowledge of the sources of degradation; and a comparison of these analyses made on fabrics from different bleach processes by revealing the advantages and limitations of each process, should, in turn, result in improved and less costly procedures and a longer life of the fabrics.

Of the more than 10.7 billion yards of woven fabrics produced in the United States during 1945 (*28*)[3] bleached cotton fabrics accounted for 6.6 billion yards (47 yards per inhabitant). In view of the importance of bleaching in the purification of cotton, the Southern Regional Research Laboratory of the United States Department of Agriculture made an investigation of the chemistry of the various bleaching operations as applied commercially, for the purpose of improving the processes and raising the quality of the resultant fabric.

The part of that broad program which is reported here is the survey of a number of representative modern commercial bleach processes. The report includes a comparison of the different methods; the determination of what is accomplished in each step, and of the characteristics or quality of acceptable or marketable fabrics; and, finally, a discussion of some measurable properties of the fabrics, especially those which indicate deterioration. Such an impartial survey should contribute toward the understanding and possible solution of many problems in the finishing of cotton fabrics.

LITERATURE REVIEW

Although considerable literature has accumulated regarding various aspects of the kier boiling and bleaching of cotton yarns and fabrics, the evaluation of procedures by comparison of extensive cloth analyses has been rare. Scholefield and Ward (*25*) compared the whiteness, extractable matter, wettability, copper number, and tensile strength of a few fabrics that had been kier boiled and bleached both with and without the use of a wetting agent (Lissapol A), as well as with and without the use of a rosin soap. They recommended use of the wetting agent.

Hall (*13*) (using a translation) compares a Russian process described by Filipov and Voronkov (*12*) with the usual American procedures. He lists the percentages of fats and waxes, nitrogen, and ash found in bleached fabrics and gives a table of capillarities or absorbencies measured by the rate water ascends a strip of fabric suspended vertically so that a portion remains above the liquid. Kuchinka (*21*) used Nodder's alkali solubility number (*23*) to evaluate damage to linens by alkaline hydrogen peroxide and pointed out that the increase of the solubility number of cotton cellulose, just as for flax cellulose, is approximately proportional to the strength of the degradation reactions which caused the increase.

A report by Hebden (*14*) of his investigation "to determine what constitutes a good bleach from a chemical standpoint" gives probably the best published analyses of cotton cloth showing quantities of residual impurities as well as the quantities of impurities removed during the bleaching operations. Hebden determined carbon, hydrogen, oxygen, nitrogen (and protein), and phosphorus contents;

[3] Italic numbers in parentheses refer to Literature Cited, p. 35.

and the ether and alcohol extracts of gray goods and the same materials after a steep, first boil, second boil, chemic, and sour. His data, presented in tabular form, showed progressive changes in the fabrics.

An anonymous article (*1*) in 1941 charts changes in tensile strength of gray cloth at the following stages of manufacture: Desized, boiled out, dyed, printed, and finished. No similar article listing comparative chemical properties of fabrics as they pass through a modern bleachery has been found.

CONSTITUENTS REMOVED

Each cotton fiber is a tubular ribbon-like cell, from 1,000 to 4,000 times as long as it measures in cross section, whose chemical composition reflects its cellular nature. Although cellulose is its major constituent, any of the substances commonly found in plant cells may be expected to be present in at least small amounts.

Guthrie and his coworkers [4] have assembled from the literature the selected values for the composition of a typical cotton (table 1).

TABLE 1.—*Composition of typical mature cotton fiber*

Constituent	Dry weight		
	Typical	Low	High
	Percent	*Percent*	*Percent*
Cellulose	94.0	88.0	96.0
Protein (N×6.25)	1.3	1.1	1.9
Pectic substances	1.2	.7	1.2
Ash	1.2	.7	1.6
Wax	.6	.4	1.0
Total sugars	.3		
Pigments	Trace		
Other	1.4		

Of the constituents which it is desired to remove from fibers, yarns, or fabrics during bleaching, the most troublesome are the seed-coat fragments and aborted seeds, which the practical bleacher calls "motes." These fragments often cling so tenaciously to the lint that they resist all mechanical cleansing and appear as dark brown or black specks in the yarn or woven gray cloth.

Besides the troublesome natural constituents of cotton, other materials which have been added by manufacturers for various purposes must also be removed during bleaching. Some manufacturers add approximately 0.25 percent (weight basis) mineral oil in the opening or in the pickers, in an effort to reduce fly and dust, produce a cleaner environment, increase flexibility in mill production, and prevent breakage of the long, usable fibers. The warp yarns almost always contain a sizing put there to add abrasion resistance and strength and thereby reduce the number of yarn breaks in the weaving operations.

[4] GUTHRIE, J. D., HOFFPAUIR, C. L., STEINER, E. T., and STANSBURY, M. F. SURVEY OF THE CHEMICAL COMPOSITION OF COTTON FIBERS, COTTONSEED, PEANUTS, AND SWEETPOTATOES. A LITERATURE REVIEW. U. S. Bur. Agr. and Indus. Chem., AIC-61. 85 pp. 1944. [Mimeographed.]

In cotton materials, this sizing is usually composed of starch and a softening agent.

The usual enzyme steep and alkaline kier boil to which cotton fabrics are subjected probably removes all the sizing materials, pectins, and protein residues, about 95 percent of the ash, about 70 percent of the wax, and almost all the "motes" and pigments. The removal of these materials is thought to be accomplished by a saponification and by an emulsification. A very probable action (*27*) of the proteins in the kier is to form amino acids that are responsible for some of the emulsification of those constituents that resist saponification and which give the characteristic color and odor to the kier liquors. The amino acids, moreover, appear to react with sodium hypochlorite (*20*) and assist in the bleaching effect.

MILL PROCESSES

The 11 mill processes investigated represent common adaptations of three general bleaching procedures: The caustic soda kier boil and hypochlorite bleach; the caustic soda kier boil and peroxide kier bleach; and the continuous processes using hydrogen peroxide or chlorite. These representative bleaching processes are classified and described below.

Class I: Caustic Soda Kier Boil and Hypochlorite Bleach

(Processes Nos. 1–4)

The caustic soda kier boil and hypochlorite bleach procedures (fig. 1), developed empirically, consist of subjecting the materials to an

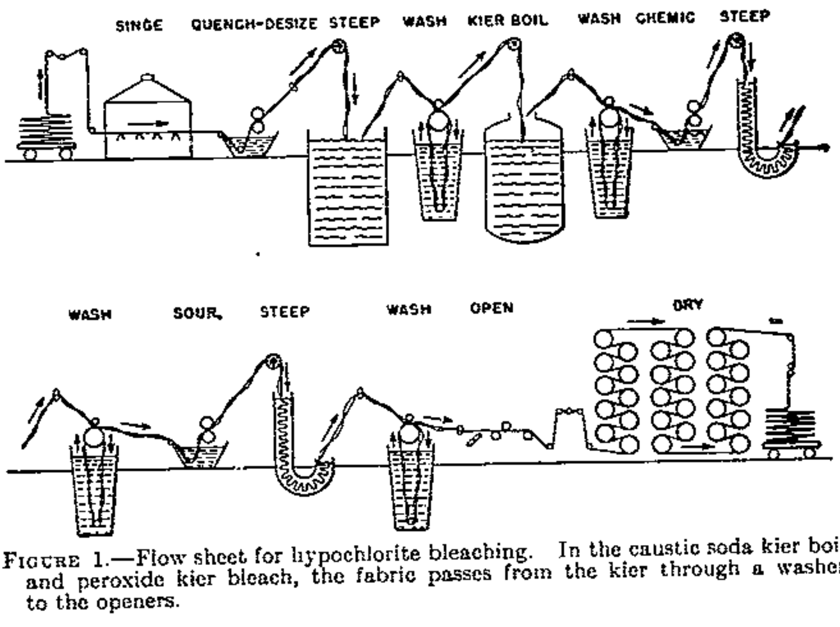

Figure 1.—Flow sheet for hypochlorite bleaching. In the caustic soda kier boil and peroxide kier bleach, the fabric passes from the kier through a washer to the openers.

alkali boil under pressure, or the kier boil, followed by treatment with sodium hypochlorite at ordinary temperature and pressure, which is the true bleach.

The kier, in general, consists of a cylindrical shell, with rounded ends, having a capacity of 1 to 5 tons of cloth, equipped with a false bottom, a manhole in the top, and a circulating system for the scouring solutions. The cloth is loaded into the kier through the manhole, chained in place, and covered with the kier solution. In operation, a heavy iron cover closes the manhole, while an outside pump and heating system forces the hot solution through a spray head over the cloth. The whole boil is conducted under pressure, usually of 15 pounds.

The hypochlorite bleach consists of passing the cloth through the solution containing sodium hypochlorite, squeezing out the excess, and piling in a bin until the nascent oxygen liberated decolorizes the cloth.

The entire process is, of course, quite intricate, the details varying with local conditions, water supply, chemicals available, and the design, style, and degree of purification demanded by the end use of the fabrics. For instance, materials intended for dyeing may be given an abbreviated process (No. 4).

Process No. 1

1. Wet with sulfuric acid (0.75° Twad. or 0.7 percent) for 5 minutes.
2. Wash without steeping.
3. Plait into the kier with automatic piler. Add the kier formula (0.34 percent tetrasodium pyrophosphate, 3 percent sodium hydroxide, both computed on the weight of the goods), and sufficient water to make a 3 to 1 solution to cloth ratio,[5] and boil for 8 hours (measured from the time full pressure is reached) at 15 pounds' pressure. The kier remains filled well above the cloth during the boil.
4. Wash down in the kier and pull from the kier through a washer.
5. Bleach by passing through sodium hypochlorite (1.5° to 2° Twad. or 1.875 pounds of chlorine per 1,000 pounds of cloth), squeezing to approximately 100 percent retention and piling in a bin for 3 to 4 hours.
6. Wash and pass through sulfuric acid (0.75° Twad. or 0.7 percent).
7. Wash and transfer to the white bins.

Process No. 2

1. Wet with 2- to 3-percent malt diastase at 140° F. and pile in a bin for 2 to 3 hours. (The temperature remains near 140° during storage.) Wash after the steeping.
2. Pass through sulfuric acid (2° Twad. or 1.5-percent) and wash again, without steeping.
3. Plait into the kier with automatic piler. Neutralize excess acid with sodium carbonate in the kier and boil for 10 hours (approximately 8 hours after full pressure is reached) at 15 pounds' pressure in 3.6 percent caustic soda (on weight of the goods) and enough water to make a 3 to 1 (weight basis) solution to cloth ratio.

[5] Solution to cloth ratios are computed on the assumption that the cloth carries its own weight of water into the kier.

4. Wash thoroughly in the kier and pull from the kier through a washer.

5. Bleach by passing through sodium hypochlorite (0.75° Twad. or 1.875 pounds of chlorine per 1,000 pounds of cloth), squeezing to approximately 100 percent retention, and piling in a bin for about 1 hour.

6. Wash and antichlor with approximately 0.1 percent sodium bisulfite.

7. Wash and transfer to the white bins.

Process No. 3

1. Singe, quench in hot water (near to boiling), and pile in bins for 4 hours.

2. Wash with cold water; squeeze to about 100 percent retention.

3. Plait into kier by hand, and boil for 12 to 14 hours (measured from the time the lid goes on, or about 10 to 12 hours after full pressure is reached), at 28 to 30 pounds' pressure in a kier solution made according to the following formula: 3 percent caustic soda, 1.5 percent sodium silicate, 0.25 percent kier assistant (long-chain alcohol sulfate), all based on the weight of the cloth, and enough water to make a 3 to 1 (weight basis) solution to cloth ratio.

4. Drain the kier, wash down thoroughly in the kier, and pull from the kier through a washer.

5. Bleach by passing through sodium hypochlorite, maintained at 1 gm. of available chlorine per liter of solution, approximately 100 percent retention, and piling in a bin for 0.5 hour.

6. Wash and pass through sulfuric and hydrochloric acid mixture in 2 to 1 ratio (1° to 1.5° Twad. or 1.0 to 1.5 percent).

7. Wash and transfer to the white bins.

Process No. 4

1. Pass the gray cloth through a rope washer.

2. Treat with sulfuric acid (1.5° to 2° Twad., or 1.25 to 1.5 percent) at 120° F.

3. Wash with water containing sufficient caustic soda to neutralize all acid present.

4. Plait into the kier with an automatic piler and boil for 10 hours (measured from the time full pressure is reached) at 15 pounds' pressure in a solution made according to the following formula: 3 percent caustic soda, 0.33 percent S.T. Solvent (one-half pine oil and one-half sulfonated castor oil), both computed on the weight of the goods, and sufficient water to make a 3 to 1 (weight basis) solution to cloth ratio.

5. Drain the kier and wash down thoroughly in the kier.

6. Pull the cloth from the kier through sulfuric acid (0.5° Twad. or 0.5 percent).

7. Neutralize with sodium carbonate and pass through a washer into the white bins.

CLASS II: CAUSTIC SODA KIER BOIL AND PEROXIDE KIER BLEACH (PROCESSES Nos. 5–7)

The commercial production of concentrated hydrogen peroxide solutions made possible their substitution for calcium or sodium hypochlorite in bleaching. Thus, suitable solutions of hydrogen peroxide can be prepared and circulated, at elevated temperatures, through the cloth while it remains in the kier. Further treatment consists of merely washing with cold water to remove the reagents from the cloth. Generally the chemicals used are more expensive, but the fabrics receive less handling and mechanical damage. These procedures are usually reserved for the lighter weight and better grade fabrics.

The same equipment is used as for the caustic soda kier boil and hypochlorite bleach (fig. 1).

Process No. 5

1. Wet with hot water, squeeze.
2. Plait into kier, by hand, and heat 4 hours, at 180° to 200° F. in the following solution: 1.5 percent caustic soda, 0.625 percent sodium silicate, 0.625 percent detergent (N–25–C or concentrated amines and alkylaromatic sulfonates), and enough water to make a 3 to 1 solution to cloth ratio.
3. Drain, wash down thoroughly in the kier, and bleach for 8 hours (measured from the time heating starts), at 180° to 200° F. with 1-volume hydrogen peroxide, buffered to pH 11, with sodium silicate in a 3 to 1 ratio of solution to cloth.
4. Drain, wash down thoroughly in the kier, and pull from the kier through a washer into the white bins.
5. Soap wash (warm) and wash with cold water. (This step is usually omitted.)

Process No. 6

1. Wet with water and store in bins overnight (about 18 hours).
2. Pull from bins through a washer.
3. Wet with sulfuric acid (0.75° Twad. or 0.7 percent) for 5 minutes.
4. Wash without steeping.
5. Plait into the kier with an automatic piler. Add solution of the following formula: 0.33 percent tetrasodium pyrophosphate, 3 percent sodium hydroxide, both computed on the weight of the goods, and water to make 3 to 1 ratio of solution to cloth, and boil for 8 hours (measured from the time full pressure is reached) at 15 pounds pressure.
6. Wash down thoroughly in the kier and bleach in ratio of 2.7 to 1 solution to cloth, with 0.67-volume hydrogen peroxide, buffered to approximately pH 11 with sodium silicate, for 4 hours (measured from the time desired temperature is reached) at 180° F. Drain off the bleach solution and wash down in the kier.
7. Pull from the kier through a cold-water washer and into the white bins.

Process No. 7

1. Wet with 2- to 3-percent malt diastase solution at 140° F. and pile in a bin for 2 to 3 hours. (The temperature remains near 140° during storage.) Wash after the steeping.
2. Plait into the kier with an automatic piler and boil for 5 hours (approximately 3 hours after full pressure is reached) at 15 pounds' pressure in 2.5 percent caustic soda (on the weight of the goods) and enough water to make a 3 to 1 (weight basis) solution to cloth ratio.
3. Drain the kier and wash the cloth thoroughly in the kier.
4. Pull the cloth from the kier through sulfuric acid (2° Twad. or 1.5 percent) and a washer and plait into a second kier with an automatic piler.
5. Neutralize excess acid with sodium carbonate in the kier, drain, and boil for 8 hours (approximately 6 hours after full pressure is reached) at 15 pounds' pressure in 1.1 percent caustic soda (on the weight of the goods) and enough water to make a 3 to 1 solution to cloth ratio.
6. Wash down thoroughly in the kier and bleach 5 hours (approximately 4 hours after the desired temperature is reached) at 180° F. with 0.5-volume hydrogen peroxide, buffered to approximately pH 11 with sodium silicate, in a 3 to 1 solution to cloth ratio.
7. Wash down thoroughly in the kier and pull the cloth from the kier through a washer into the white bins.

Class III: Continuous Bleaching (Processes Nos. 8–11)

The development of continuous bleach processes materially shortened the time required for the fabrics to pass through the bleachery. The fabrics are singed, desized, and passed through the continuous bleach unit (fig. 2), which consists of passing them through the caustic saturator, J-box (at approximately 200° F.), washer, squeeze rolls, hydrogen peroxide saturator, J-box (at approximately 180°), and washer; and are then transferred to the white bins.

Continuous processes are time-saving, give a continuous even flow of fabrics from the gray room to the white bins; occupy less floor space than the batch processes; and require fewer operators. The equipment, however, is more costly and careful controls of all operations are required.

Process No. 8

1. Pass through sulfuric and hydrochloric acid mixture in 2 to 1 ratio (diluted to 1 to 1.5° Twad. or 1 to 1.5 percent), allowing 120 percent retention (weight basis), and store in bin 2.5 hours.
2. Wash thoroughly to remove the acid.
3. Pass through saturator allowing 100 percent retention of caustic solution (3 percent caustic soda, based on the weight of the goods).
4. Pass through steam saturator and steep in J-box at 180°–203° F. for 1 hour.
5. Wash with cold water.
6. Pass through saturator, allowing 100 percent retention (weight basis) of 1 volume hydrogen peroxide solution buffered to approximately pH 11 with sodium silicate.

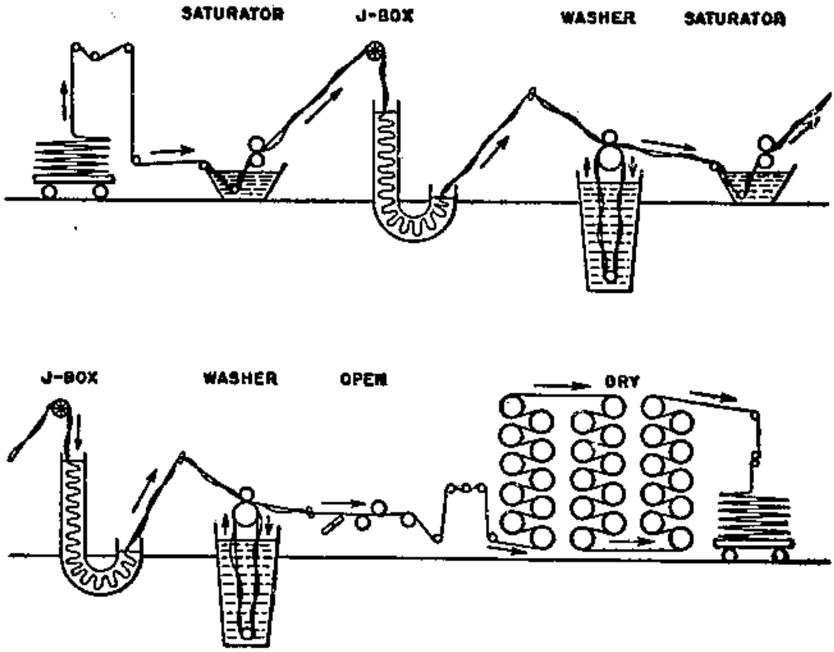

FIGURE 2.—Flow sheet for continuous bleaching. In one of the two commercially available continuous-bleaching installations, steam is injected into the J-box, while in the other installation, the steam is injected into the fabric just before it enters the J-box.

7. Pass through steam saturator and steep in J-box at 180°–203° F. for about 1 hour and 20 minutes.
8. Wash thoroughly and transfer to the white bins.

Process No. 9

1. Pass the gray cloth through sulfuric acid (0.5° Twad. or 0.5 percent).
2. Wash with cold water.
3. Pass through caustic saturator allowing 100 percent retention (weight basis) of 3 percent caustic soda.
4. Pass through steam saturator and steep in J-box for 1 hour ± 1 minute at 210°–212° F.
5. Wash with cold water.
6. Pass through peroxide saturator allowing 100 percent retention of 1 volume hydrogen peroxide buffered to pH 11 to 11.4 with sodium silicate.
7. Pass through steam saturator into J-box steep for 1 hour ± 1 minute at 160°–170° F.
8. Wash and transfer to the white bins.

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Process No. 10

1. Pass through sulfuric and hydrochloric acid mixture in 2 to 1 ratio (diluted to 1° to 1.5° Twad. or 1 to 1.5 percent), allowing 120 percent retention (weight basis), and store in a bin 45 minutes.
2. Wash thoroughly to remove the acid.
3. Pass through saturator where cloth retains 100 percent (weight basis) of 1 volume hydrogen peroxide buffered to approximately pH 11 with sodium silicate.
4. Pass through steam saturator and steep in J-box at 180°–203° F. for 1 hour.
5. Wash with cold water.
6. Pass through saturator containing hydrogen peroxide, prepared according to the formula used in step 3.
7. Pass through steam saturator and steep in J-box at 180°–203° F. for 1 hour.
8. Wash thoroughly and transfer to the white bins.

Process No. 11

1. Pass the gray cloth through sulfuric acid (2° Twad. or 1.5 percent) at 95° F.
2. Wash with cold water.
3. Pass through caustic saturator, 4.5 percent caustic soda, approximately 100 percent retention.
4. Pass through steam saturator and steep in J-box for 1 hour and 25 minutes at 180° F.
5. Wash in cold water.
6. Pass through saturator permitting 100 percent retention of combined sodium hypochlorite ($NaClO$) and chlorite ($NaClO_2$) solution containing 0.71 gm. of available chlorine per liter, pH 9.8.
7. Pass through stream saturator and steep in J-box at 160°–170° F. for 1 hour and 20 minutes.[6]
8. Wash and transfer to the white bins.

MATERIALS

The two cotton fabrics used for this investigation are described as follows:

(1) A gray premium quality print cloth, 38.5 inches wide, having 64 warp and 56 filling yarns to the inch, and weighing 2.76 ounces per square yard, or 5.80 square yards per pound.

(2) A gray broadcloth, 38.5 inches wide, having 136 warp and 64 filling yarns to the inch, and weighing 4.12 ounces per square yard, or 3.88 square yards per pound.

Print cloth was used for processes Nos. 1 to 6, and 8 to 11, inclusive. A 62-yard piece was used for each process. The first 2 yards was held as a control and the balance (60 yards) was then sewed into the

[6] Owing to unavoidable circumstances, the chlorite manufacturer's recommended procedure was not followed. The results obtained therefore do not prejudice use of the material. When time permits, this part of the work will be repeated according to manufacturer's recommended procedure and the results reported.

middle of a lot of cloth just before the lot was put through each of the ten processes included. Samples approximately 12 yards long were taken after each of the principal operations.

Broadcloth was used for process No. 7. A 14-yard piece was used. Two yards were held as a control and the balance was sewed into the center of a lot of broadcloth just before it was processed. Samples approximately 2 yards long were taken after each of the principal operations.

Detailed identification of all samples is given in table 2.

TABLE 2.—*Detailed identification of fabric samples for analysis*

Sample No.	Process[1] No.	Taken after operation No.	Treatments
538	1	Control	None.
539	1	2	Sour.
540	1	4	Sour, kier boil.
541	1	7	Sour, kier boil, hypochlorite bleach, air dry.
542	1	7	Same as 541 but given water mangle and dry cans.
545	2	Control	None.
546	2	1	Desize (enzyme).
547	2	2	Desize, sour.
548	2	4	Desize, sour, kier boil.
549	2	7	Desize, sour, kier boil, hypochlorite bleach, air dry.
550	2	7	Same as 549 but given water mangle and dry cans.
587	3	Control	None.
588	3	2	Singe.
589	3	4	Singe, kier boil.
590	3	7	Singe, kier boil, hypochlorite bleach, air dry.
591	3	7	Same as 590 but given water mangle and dry cans.
559	4	Control	None.
560	4	1	Wash.
561	4	3	Wash, sour.
562	4	5	Wash, sour, kier boil.
563	4	7	Wash, sour, kier boil, sour, sodium carbonate, air dry.
564	4	7	Same as 563 but given water mangle and dry cans.
552	5	Control	None.
553	5	1	Hot water.
554	5	4	Hot water and peroxide kier bleach.
555	5	5	Hot water, peroxide kier bleach, soap wash, air dry.
556	5	5	Same as 555 but given water mangle and dry cans.
566	6	Control	None.
567	6	2	Steep.
568	6	4	Steep, sour.
569	6	7	Steep, sour, peroxide kier bleach, air dry.
570	6	7	Same as 569 but given water mangle and dry cans.
607	7	Control	None.
613	7	1	Desize (Enzyme).
614	7	3	Desize, kier boil.
615	7	4	Desize, kier boil, sour.

[1] See text for detailed description of these processes.

TABLE 2.—*Detailed identification of fabric samples for analysis*—Con.

Sample No.	Process[1] No.	Taken after operation No.	Treatments
616	7	7	Desize, kier boil, sour, kier boil, air dry.
617	7	7	Same as 616 but given water mangle and dry cans.
580	8	Control	None.
581	8	2	Sour (2½ hours).
582	8	5	Sour, caustic steep.
583	8	8	Sour, caustic steep, peroxide steep, air dry.
584	8	8	Same as 583 but given water mangle and dry cans.
602	9	Control	None.
603	9	2	Sour.
604	9	5	Sour, caustic steep.
605	9	8	Sour, caustic steep, peroxide steep, air dry.
606	9	8	Same as 605 but given water mangle and dry cans.
594	10	Control	None.
595	10	2	Sour.
596	10	5	Sour, peroxide steep.
597	10	8	Sour, peroxide steep, peroxide steep, air dry.
598	10	8	Same as 597 but given water mangle and dry cans.
573	11	Control	None.
574	11	2	Sour.
575	11	5	Sour, caustic steep.
576	11	8	Sour, caustic steep, chlorite steep, air dry.
577	11	8	Same as 576 but given water mangle and dry cans.

[1] See text for detailed description of these processes.

METHODS OF EVALUATING FABRICS

A comparison of the analyses of the gray goods with samples of the same fabrics taken after successive stages of the purification process shows the residual impurities and, by difference, the amounts of extraneous materials removed from the fabrics. To be of value, however, this information must be supplemented by measures of the changes which have occurred in the fabrics, the degree of whiteness produced, and the resulting absorbability.

The residual materials of any particular sample may be measured by its moisture content, alcohol solubility, and wax, ash, and cellulose contents. These values are usually expressed as a percentage of the oven-dry weight of sample. Fabric changes are measured by thread counts, weights per square yard, or square yards per pound, thicknesses, breaking strengths, and fluidities of the cellulose in cuprammonium hydroxide.[7]

[7] Copper numbers, methylene blue absorption, and nitrate viscosity, or fluidity indicate the nature of degradation rather than its extent. These measurements have therefore been omitted from the analyses of the processed fabrics in this investigation.

The degree of whiteness, measured in ICI (International Commission on Illumination) units, is compared to magnesium oxide white. Absorbability is measured by the "drop-square" and capillary rise methods.

Residual Materials

All samples were dried so that they contained considerably less than the equilibrium moisture content. Moistures were determined in aluminum moisture dishes on 0.5-gm. samples, weighed in an air-conditioned laboratory at 70° F. and 65 percent relative humidity (*5*) after conditioning overnight. The samples were dried to constant weight in an air oven at 105°±1° C. Regains were calculated from moisture data.

Alcohol solubility is the percentage (on dry weight of sample) of extractives obtained after 6 hours' Soxhlet extraction of 5-gm. samples. The extracts were transferred, while still hot (above 55° C.), to 100-ml. beakers, and dried by heating in an air oven at 105°±1° C. for 30 minutes, cooled in a desiccator, and weighed. This heating and weighing was repeated until two successive weighings agreed within 1.0 mg.

The amount of wax present in the cloth was determined by the Conrad method (*9*), except that 100 ml. of distilled water was used in the separatory funnels instead of the 75 ml. specified—since, with this quantity of water, phase separations occur more readily. The Conrad technique was chosen because it has been found to be more efficient than any other method (*19*) tried for extraction of waxy materials from cottons.

The ash content of the cloth was determined by the method of Fargher and Probert (*11*). Five-gram samples in silica dishes were heated to 750° C. in a muffle furnace equipped with thermostatic control and were maintained at that temperature for 2 hours. The crucibles were cooled first to approximately 400° C. in the muffle furnace, and finally in a desiccator over calcium chloride, and weighed.

Cellulose contents were determined by wet oxidation with acid-dichromate solutions, described by Kettering and Conrad (*18*), after the monoethanolamine extraction recommended by Reid, Nelson, and Aronovsky (*24*).

Fabric Changes

Thread counts are counts of the "ends" and "picks" per inch, made with a Suter pick counter equipped with a broad-field lens and traveling needle. Each recorded value is the average of 10 counts made according to the procedure recommended by A. S. T. M. (American Society for Testing Materials) (*3*).

Shrinkage was computed from the change in the number of yarns per inch using the formula:

$$\text{Percent shrinkage}=\left[1.00-\left(\frac{Wo}{W}\times\frac{Fo}{F}\right)\right]\times 100$$

where Wo is the warp thread count of the control
W is the warp thread count of the sample
Fo is the filling thread count of the control
F is the filling thread count of the sample.

A minus shrinkage represents an increase in yardage.

Weights per square yard or square yards per pound were determined by measuring a piece of each sample and weighing it under standard conditions (*5*) and computing the values by the method outlined in the A. S. T. M. procedure (*3*).

Thicknesses were measured with a dead-weighted-type gage equipped with a dial and presser foot to meet A. S. T. M. specifications (*4*). Each recorded figure is the average of 10 readings taken by the A. S. T. M. method (*3*).

Breaking strengths were made by the ravelled-strip method (*3*). Strips were cut 6 inches long by 1.25 inches wide, ravelled to 1 inch (determined by counting the threads), and broken on a pendulum-type Scott tester, between 3-inch jaws set 3 inches apart, the lower jaw traveling downward at the rate of 12 inches per minute. Each recorded figure is the average of 10 determinations.

Fluidity in cuprammonium hydroxide was measured by using the Conrad velocity gradient technique (*8*). The cuprammonium hydroxide was stored at approximately 40° F., the viscometers were filled, the cotton dissolved and time of flow readings were taken at constant temperature in an atmosphere of nitrogen. The results were computed on the basis of cellulose content and interpolated logarithmically to a mean velocity gradient of 500 reciprocal seconds (500 sec. $^{-1}$) for comparison.

Degree of Whiteness

The degree of whiteness was measured on the Hunter reflectometer (*16*). This instrument, sensitive enough to differentiate samples visually identical, employs a null method and a galvanometer to indicate equality of currents generated by two photocells which are activated by the light beams, one from the sample and the other from the light source. Photometric adjustments are measured on direct-reading scales. The method outlined by National Bureau of Standards Circular C429 (*17*), measures apparent reflectance as a function of wave length in the ICI trichromatic coefficients of the spectrum by the use of filters calibrated with magnesium oxide. A whiteness of 1.00 is pure magnesium oxide white, which has a corresponding zero yellowness.

Absorbability

The degree of absorbability was measured by the time-honored "drop-square test" (*15*), (*26*) as well as by the Weirick capillary rise method (*26*), (*29*). The drop-square test consisted of dropping a 2- × 2-inch square of the fabric on the surface of distilled water from a height of 1.5 inches and measuring with a stop watch the time required to sink. Each recorded figure is the average of 5 or more determinations. The capillary rise method consisted in measuring the height to which water rises in 10 minutes in a 1-inch strip hung vertically so that the end is immersed in the water. Readings were taken with a stop watch by the aid of a fluorescent light hung behind the sample. The eosin dye was unnecessary with the powerful fluorescent light. Warp and filling strips were measured, and, in each case, the average of 5 determinations is reported.

DISCUSSION OF RESULTS

The results will be discussed from the standpoint of residual materials in the fabrics, fabric changes produced, the resulting whitenesses, and accompanying absorbabilities.

Residual Materials

Data from analyses for residual materials are shown in tables 3 to 5 and summarized in table 6.

Table 3.—*Residual materials found in fabric samples taken after progressive operations of the caustic soda kier boil and hypochlorite bleach*

Sample No.	Description	Moisture	Alcohol-soluble material[1]	Wax[1]	Ash[1]	Cellulose[1]
		Percent	*Percent*	*Percent*	*Percent*	*Percent*
	Process No. 1:					
538	Control	6.76	2.05	1.00	1.30	91.25
539	Gray sour	7.66	1.14	1.10	.20	93.06
540	Gray sour, kier boil	6.96	.50	.48	.17	99.48
541	Same plus hypochlorite bleach, sour, air-dried	6.80	.46	.42	.02	99.38
542	Same as 541 but given water mangle and dry cans	6.30	.42	.42	.00	99.50
	Process No. 2:					
545	Control	6.84	1.98	1.03	1.26	90.23
546	Desize, wash	7.36	1.60	1.28	.26	96.06
547	Desize, wash, sour, wash	7.28	1.24	1.12	.23	96.26
548	Same plus kier boil, wash	6.98	.40	.34	.02	96.82
549	Same plus hypochlorite bleach, air-dried	6.99	.34	.28	.02	98.97
550	Same as 549 but given water mangle and dry cans	6.64	.32	.28	.02	98.42
	Process No. 3:					
587	Control	6.86	1.92	1.02	1.25	92.18
588	Singe, quench, wash	7.65	1.11	1.02	.28	90.03
589	Singe, quench, wash, kier boil, wash	6.82	.55	.52	.25	98.46
590	Same plus hypochlorite bleach, sour, wash, air-dried	6.78	.40	.43	.08	99.01
591	Same as 590 but given water mangle and dry cans	6.54	.43	.46	.08	98.86
	Process No. 4 (no bleach):					
559	Control	6.89	1.99	1.02	1.24	90.85
560	Water wash	7.78	1.47	1.38	.50	89.52
561	Water wash, sour, wash	7.64	1.52	.96	.68	94.93
562	Same plus kier boil, washed in kier	6.62	.56	.52	.02	98.68
563	Same plus sour, sodium carbonate, air-dried	6.67	.44	.34	.07	98.86
564	Same as 563 but given water mangle and dry cans	6.30	.38	.34	.07	99.14

[1] The values are based on oven-dry weights of the fabrics. Wax is part of the alcohol-soluble materials.

TABLE 4.—*Residual materials found in fabric samples taken after progressive operations of the caustic soda kier boil peroxide kier bleach process*

Sample No.	Description	Moisture	Alcohol-soluble material[1]	Wax[1]	Ash[1]	Cellulose[1]
		Percent	*Percent*	*Percent*	*Percent*	*Percent*
	Process No. 5:					
552	Control	6.90	1.97	0.99	1.22	92.04
553	Hot water, cold water dip	7.46	1.62	1.04	.90	90.62
554	Hot water, cold water dip, peroxide kier bleach	6.92	1.02	.85	.42	98.06
555	Same plus soap wash, water wash, air-dried	6.51	.78	.69	.24	97.25
556	Same as 555 but given water mangle and dry cans	6.06	.80	.77	.28	97.34
	Process No. 6:					
566	Control	6.85	1.94	1.00	1.26	92.11
567	Steep	6.73	1.65	1.06	.36	95.81
568	Steep, gray sour, wash	6.76	1.10	1.04	.14	96.10
569	Same plus peroxide kier bleach, air-dried	6.55	.36	.30	.13	99.56
570	Same as 569 but given water mangle and dry cans	6.19	.36	.36	.10	99.34
	Process No. 7:					
607	Control	7.08	2.05	1.32	.74	93.70
613	Desize, wash	6.68	1.74	1.41	.24	95.53
614	Desize, wash, kier boil	6.50	.83	.76	.16	97.76
615	Desize, wash, kier boil, sour, wash	6.72	.44	.49	.04	97.85
616	Same as 615 and kier boil, peroxide kier bleach, wash, air-dried	6.67	.26	.26	.05	98.34
617	Same as 616 but given water mangle and dry cans	5.85	.42	.34	.12	97.66

[1] The values are based on the oven-dry weights of the fabrics. Wax is part of the alcohol-soluble materials.

TABLE 5.—*Residual materials found in fabric samples taken after progressive operations of the continuous-bleach processes*

Sample No.	Description	Moisture	Alcohol-soluble material[1]	Wax[1]	Ash[1]	Cellulose[1]
	Process No. 8:	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
580	Control	6. 84	1. 94	1. 08	1. 14	91. 84
581	Sour (2.5 hours in acid), wash	6. 88	1. 17	1. 02	. 36	92. 42
582	Sour, wash, caustic, steam, J-box, wash	6. 16	1. 02	. 79	. 40	98. 50
583	Same plus peroxide, steam, J-box, wash, air-dried	6. 44	. 66	. 60	. 15	98. 56
584	Same as 583 but given water mangle and dry cans	6. 08	. 66	. 60	. 14	98. 74
	Process No. 9:					
602	Control	7. 02	1. 98	1. 00	1. 22	90. 79
603	Sour, wash	7. 40	1. 16	1. 00	. 33	92. 15
604	Sour, wash, caustic, saturator, steam, J-box, wash	6. 72	. 86	. 68	. 06	98. 99
605	Same plus peroxide saturator, steam, J-box, wash, air-dried	6. 58	. 46	. 38	. 06	99. 57
606	Same as 605 but given water mangle and dry cans	6. 42	. 46	. 36	. 06	98. 77
	Process No. 10:					
594	Control	7. 04	1. 99	1. 00	1. 26	91. 46
595	Sour (45 minutes in acid) wash	7. 13	1. 18	1. 05	. 50	91. 89
596	Sour, wash, peroxide, steam, J-box, wash	6. 19	. 76	. 82	. 29	97. 72
597	Same plus second peroxide, steam, J-box, air-dried	6. 06	. 54	. 52	. 22	96. 96
598	Same as 597 but given water mangle and dry cans	5. 96	. 55	. 56	. 18	98. 48
	Process No. 11:					
573	Control	6. 89	1. 92	1. 04	1. 24	92. 19
574	Sour	7. 52	1. 28	1. 02	. 26	93. 32
575	Sour, caustic, J-box, wash, air-dried	6. 97	. 46	. 41	. 02	98. 67
576	Sour, caustic, J-box, chemic, J-box, air-dried	6. 86	. 42	. 36	. 05	98. 32
577	Same as 576 but given water mangle and dry cans	6. 68	. 44	. 36	. 04	98. 20

[1] The values are based on the oven-dry weights of the fabrics. Wax is part of the alcohol-soluble materials.

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TABLE 6.—*Summary of analyses of print cloth samples for residual materials after the various steps of three methods of bleaching*

Treatment	Moisture from standard atmosphere	Alcohol-soluble materials[1]	Wax[1]	Ash[1]	Cellulose[1]	Other materials by difference[1]
Hypochlorite bleach: (Processes 1–4):	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Control	6. 82	1. 98	1. 02	1. 27	91. 22	5. 53
Desized	7. 53	1. 16	1. 08	. 25	93. 12	5. 47
Kier boiled	6. 92	. 48	. 45	. 22	98. 25	1. 05
Bleached	6. 54	. 39	. 39	. 03	98. 93	. 65
Peroxide kier bleach (Processes 5–7):						
Control	6. 88	1. 98	1. 00	1. 24	92. 08	4. 70
Desized	7. 10	1. 57	1. 05	. 63	93. 21	4. 59
Bleached	6. 12	. 59	. 56	. 19	98. 34	. 68
Continuous bleach (Processes 8–11):						
Control	6. 97	1. 97	1. 03	1. 21	91. 36	5. 46
Desized	7. 14	1. 17	1. 02	. 40	92. 15	6. 28
First steep	6. 36	. 88	. 76	. 25	98. 40	. 47
Bleached	6. 15	. 55	. 51	. 13	98. 66	. 68
Average:						
Control	6. 88	1. 98	1. 02	1. 24	91. 62	5. 33
Desized	7. 28	1. 32	1. 06	. 43	92. 91	5. 38
Bleached	6. 28	. 51	. 46	. 13	98. 66	. 67

[1] The values are based on the oven-dry weights of the fabrics. Wax is part of the alcohol-soluble materials.

Moisture content.—The moisture content of fibers is influenced by the amounts of extraneous materials present; the amount of exposed fiber surfaces; the number, size, and shape of fiber capillaries or intermiscellar spaces; and by the previous history of moisture relations of the fibers. Moisture content is also obviously influenced by the temperature and relative humidity of the surrounding atmosphere; but in this investigation these need not be considered, since all samples were conditioned in the same manner and weighed in a standard atmosphere. The majority, if not all, of the variations found in moisture content, therefore, indicate differences in fibers or fabrics.

Treatments used to wet out the fabrics or remove the warp sizing (table 7) increased moisture content except in processes Nos. 6 and 7. Moreover, the increases were too large to be attributed to fluctuations in temperature, relative humidity, or other inaccuracies in the analyses. It appears probable that a combination of phenomena acting simultaneously, rather than the action of any one phenomenon, so altered the fibers that changed moisture affinities resulted.

TABLE 7.—*Effect of wetting, souring, or desizing treatments on moisture and wax contents of fabrics*

Process No.	Treatment	Moisture		Wax	
		Control	Desized	Control	Desized
		Percent	*Percent*	*Percent*	*Percent*
1	Gray sour (cold)	6.76	7.66	1.00	1.10
2	Malt diastase, 2–3 percent, 140° F	6.84	7.36	1.03	1.28
3	Singe, quench, and pile in bin	6.86	7.65	1.02	1.02
4	Gray sour, cold water dip, and squeeze	6.89	7.64	1.02	1.38
5	Hot water dip	6.90	7.46	.99	1.04
6	Hot water dip; pile in bin 18 hours	6.85	6.76	1.00	1.06
7	Malt diastase, 2–3 percent, 140° F	7.08	6.68	1.32	1.41
8	Gray sour (2.5 hours in acid)	6.84	6.88	1.08	1.02
9	Acid dip and cold water dip	7.02	7.40	1.00	1.00
10	Gray sour (0.75 hour in acid)	7.04	7.13	1.00	1.05
11	Gray sour, 95° F., cold water wash	6.89	7.52	1.04	1.02

Alcohol solubility and wax analyses.—Because of the exceptional power of alcohol to remove a variety of constituents from cotton, including the waxes, sugars, nitrogeneous or protein residues, and a large part of the mineral salts, a reduction in alcohol solubility indicates better purification.

Wax analyses perhaps provide the best indication of the effectiveness of purification treatments. Cotton contains a considerable number of true waxes and resins which resist saponification so strongly that removal is difficult if not impossible.

Based on results obtained in analyzing fabrics for alcohol-soluble materials and wax (table 6) the caustic soda kier boil and hypochlorite bleach process is superior to either the caustic soda kier boil and peroxide kier bleach or the continuous processes.

The wetting-out and desizing treatments (table 7) appreciably increased wax content in processes Nos. 2 and 7; and reduced it in only one process, No. 11—so slightly, however, that the reduction cannot be considered significant. Malt diastase, used as the desizing agent in both cases, apparently deposits a small amount of wax on the fabrics. In processes Nos. 5 and 6, a slight apparent increase in wax content after hot water treatments probably was due to the use of hard water (table 8) and is not significant.

TABLE 8.—*Effect of soft and hard water, washer type, and kier or bleach assistant on wax removal from commercially bleached fabrics*

Process No.	Type of water	Type of rope washer	Kier or bleach assistants	Wax content based on oven-dry weight		
				Control	Bleached, air dried	Bleached, can dried
				Percent	*Percent*	*Percent*
1	Hard	12-strand tight	Tetrasodium pyrophosphate	1. 00	0. 42	0. 42
2	Soft	12-strand loose	None	1. 03	. 28	. 28
3	Hard	8-strand loose	Sodium silicate, long chain alcohol sulfate	1. 02	. 43	. 46
4	Soft	24-strand tight	Pine oil-sulfonated castor oil	1. 02	. 34	. 34
5	Hard	8-strand loose	Sodium silicate, amines and alkylaryl aromatic sulfates	. 99	. 69	. 77
6	do	12-strand tight	Tetrasodium pyrophosphate	1. 00	. 30	. 36
7	Soft	12-strand loose	None	1. 32	. 26	. 34
8	Hard	8-strand loose	Sodium silicate	1. 08	. 60	. 60
9	do	24-strand tight	Sodium silicate	1. 00	. 38	. 36
10	do	8-strand loose	Sodium silicate	1. 00	. 52	. 56
11	Soft	24-strand tight	None	1. 04	. 36	. 36

Wax contents of the bleached fabrics were higher than expected from data in the literature (*2, 7, 22*). The comparisons in table 8 leave little doubt that high wax values are associated with faulty washing procedures together with the use of hard water subsequent to kier boiling. The four processes using soft water reduced the wax content of the fabrics more than did any process using hard water. Moreover, the addition of pine oil and sulfonated castor oil (process No. 4) failed to reduce the wax content below that found when no detergent or other assistant was added at any stage in the process. The highest wax values were found when 8-strand washers were used These considerations suggest rather strongly that soft water and efficient washing procedures were responsible for removal of all but small quantities of residual wax.

Ash.—Ash determinations which measure residual mineral matter afford a good means of judging kier boiling and bleaching efficiency. In none of the processes is the ash content of bleached fabrics excessively high. The ash content data (table 6) show caustic soda kier boil and hypochlorite bleach to be again superior to either of the other two methods. Processes Nos. 2 and 4 are the most effective in removing ash. It is noteworthy that the latter process did not include any bleach.

Cellulose content.—In addition to forming the basis for calculating fluidities of cellulose in cuprammonium hydroxide, cellulose content gives a measure of the effectiveness of purification treatments, a high content indicating small quantities of residual extraneous materials. The cellulose contents found show good purification in all processes (table 6). When judged by these values, the caustic soda kier boil and hypochlorite bleach is superior to either of the other two bleaching procedures.

Fabric Changes

Data from analyses which show the changes in the fabrics that take place during bleaching are shown in tables 9 to 11 and summarized in table 12.

TABLE 9.—*Changes found in fabric samples taken after progressive operations in the caustic soda kier boil and hypochlorite bleach*

Sample No.	Description	Yarns per inch		Shrinkage	Breaking strength		Adjusted breaking strength		Thickness	Weight		Fluidity: (C=0.5 gm per 100 ml; G=500 sec.$^{-1}$)
		Warp	Filling		Warp	Filling	64 warp yarns	56 filling yarns		*Ounces per square yard*	*Square yards per pound*	
				Percent	*Pounds*	*Pounds*	*Pounds*	*Pounds*	*Inches*			*Rhes*
	Process No. 1:											
538	Control	64	56	0	46.7	27.4	46.7	27.4	0.0104	2.78	5.76	2.12
539	Gray sour	65	58	4.94	45.1	25.9	44.4	25.0	.0117	2.69	5.95	2.48
540	Gray sour, kier boil	72	58	14.18	50.8	27.7	45.2	26.7	.0120	2.59	6.18	2.60
541	Same plus hypochlorite bleach, sour, air-dried	72	58	14.18	45.7	25.8	40.6	24.9	.0116	2.63	6.08	5.98
542	Same as 541 but given water mangle and dry cans	69	55	5.56	47.9	26.2	44.4	26.7	.0102	2.41	6.64	5.74
	Process No. 2:											
545	Control	64	56	0	46.5	27.4	46.5	27.4	.0107	2.76	5.80	2.10
546	Desize, wash	70	59	13.22	43.4	23.4	39.7	22.2	.0121	2.65	6.04	2.26
547	Desize, wash, sour, wash	69	59	11.96	42.3	24.6	39.2	23.3	.0121	2.69	5.95	2.36
548	Same plus kier boil, wash	73	60	18.17	51.8	26.9	45.4	25.1	.0123	2.63	6.08	2.80
549	Same plus hypochlorite bleach, air-dried	71	58	12.97	47.9	26.9	43.2	26.0	.0122	2.64	6.06	4.86
550	Same as 549 but given water mangle and dry cans	69	55	5.56	47.5	25.0	44.0	25.5	.0105	2.42	6.61	4.83
	Process No. 3:											
587	Control	64	56	0	48.1	27.3	48.1	27.3	.0105	2.77	5.78	2.12
588	Singe, quench, wash	70	57	10.17	50.2	26.5	45.9	26.0	.0116	2.73	5.86	2.12

589	Singe, quench, wash, kier boil, wash	72	61	18.40	41.9	24.4	37.2	22.4	.0120	2.59	6.18	3.04
590	Same plus hypochlorite bleach, sour, wash, air-dried	73	60	18.18	44.2	22.6	38.8	21.1	.0116	2.67	5.99	7.00
591	Same as 590 but given water mangle and dry cans	72	57	12.67	45.6	23.2	40.5	22.8	.0112	2.55	6.27	7.17
	Process No. 4 (no bleach):											
559	Control	63	56	0	46.9	25.0	46.9	25.0	.0105	2.74	5.84	2.14
560	Wash	68	57	8.98	48.8	25.6	45.2	25.2	.0116	2.83	5.65	2.33
561	Wash, sour, wash	70	56	10.00	44.4	25.1	40.0	25.1	.0117	2.65	6.04	2.53
562	Same plus kier boil, washed in kier	73	58	16.68	47.2	23.9	40.7	23.1	.0121	2.62	6.11	3.14
563	Same plus sour, sodium carbonate, air-dried	74	58	17.81	51.9	25.9	44.2	25.0	.0121	2.64	6.06	3.11
564	Same as 563 but given water mangle and dry cans	76	55	15.60	53.0	24.2	43.9	24.6	.0112	2.54	6.30	3.19

[1] Fluidity at a concentration (C) of 0.5 gram of cellulose per 100 milliliters of cuprammonium hydroxide solution and the values adjusted to a velocity gradient ($\overline{G}$) of 500 reciprocal seconds (500 sec.$^{-1}$).

TABLE 10.—*Changes found in fabric samples taken after progressive operations of the caustic soda kier boil and peroxide kier bleach processes*

Sample No.	Description	Yarns per inch: Warp	Yarns per inch: Filling	Shrinkage	Breaking strength: Warp	Breaking strength: Filling	Adjusted breaking strength: 64 warp yarns	Adjusted breaking strength: 56 filling yarns	Thickness	Weight: Ounces per square yard	Weight: Square yards per pound	Fluidity: (C=0.5 gm. per 100 ml.; $\bar{G}$=500 sec.$^{-1}$)[1]
				Percent	*Pounds*	*Pounds*	*Pounds*	*Pounds*	*Inches*			*Rhes*
	Process 5:											
552	Control	64	56	0	47. 2	26. 5	47. 2	26. 5	0. 0106	2. 77	5. 78	2. 15
553	Hot water and cold water dip	68	58	9. 13	49. 9	27. 4	47. 0	26. 5	. 0121	2. 91	5. 50	2. 12
554	Hot water and cold water dip, peroxide bleach	70	58	11. 72	41. 6	24. 4	38. 0	23. 6	. 0117	2. 71	5. 90	3. 29
555	Same plus soap wash, water wash, air-dried	68	58	9. 13	38. 7	24. 2	36. 4	23. [illegible]	. 0117	2. 66	6. 02	3. 15
556	Same as 555 but given water mangle and dry cans	70	56	8. 57	40. 8	24. 3	37. 3	24. 3	. 0118	2. 54	6. 30	2. 72
	Process No. 6:											
566	Control	63	56	0	46. 2	27. 5	46. 2	27. 5	. 0106	2. 74	5. 84	2. 27
567	Steep	69	57	10. 30	43. 0	22. 3	39. 3	21. 9	. 0114	2. 65	6. 04	2. 39
568	Steep, gray sour, wash	71	58	14. 33	43. 8	24. 1	38. 9	23. 3	. 0115	2. 69	5. 95	3. 32
569	Same plus peroxide kier bleach, air-dried	71	58	14. 33	50. 8	26. 7	45. 1	25. 8	. 0117	2. 59	6. 18	3. 33
570	Same as 569 but given water mangle and dry cans	73	54	10. 05	52. 9	26. 4	45. 7	27. 4	. 0103	2. 56	6. 25	3. 23
	Process No. 7:											
607	Control	136	64	0	79. 3	33. 0	[2] 79. 3	[2] 33. 0	. 0114	4. 12	3. 88	2. 06
613	Desize, wash	138	65	2. 97	76. 0	33. 6	[2] 74. 9	[2] 33. 1	. 0129	3. 83	4. 18	2. 47
614	Desize, wash, kier boil	140	65	4. 35	76. 0	34. 7	[2] 73. 8	[2] 34. 2	. 0123	3. 80	4. 21	2. 54

615	Desize, wash, kier boil, sour, wash	140	67	7. 21	79. 4	34. 4	[2] 77. 1	[2] 32. 8	. 0125	3. 79	4. 22	2. 50
616	Same as 615 plus second kier boil, peroxide bleach, wash, air-dried	141	67	7. 86	80. 5	41. 2	[2] 77. 7	[2] 39. 3	. 0120	3. 77	4. 24	3. 98
617	Same as 616, but given water mangle and dry cans	141	59	−4. 63	82. 9	35. 4	[2] 80. 0	[2] 38. 4	. 0091	3. 57	4. 48	4. 17

[1] Fluidity at a concentration (C) of 0.5 gram of cellulose per 100 milliliters of cuprammonium hydroxide solution and the values adjusted to a velocity gradient ($\overline{G}$) of 500 reciprocal seconds (500 sec.$^{-1}$).

[2] Adjusted to 136 warp and 64 filling yarns to the inch.

TABLE 11.—*Changes found in fabric samples taken after progressive operations of the continuous-bleach process*

Sample No.	Description	Yarns per inch		Shrinkage	Breaking strength		Adjusted breaking strength		Thickness	Weight		Fluidity: $\left(C=0.5 \text{ gm. per } 100 \text{ ml. } \bar{G}=500 \text{ sec.}^{-1}\right)$
		Warp	Filling		Warp	Filling	64 warp yarns	56 filling yarns		Ounces per square yard	Square yards per pound	
				Percent	*Pounds*	*Pounds*	*Pounds*	*Pounds*	*Inches*			*Rhes*
	Process No. 8:											
580	Control	64	56	0	44. 5	24. 4	44. 5	24. 4	0. 0104	2. 74	5. 84	2. 16
581	Sour (2.5 hours in acid) wash	69	59	11. 76	47. 2	23. 9	43. 8	22. 7	. 0115	2. 85	5. 61	2. 59
582	Sour, wash, caustic, steam, J-box, wash	72	59	15. 63	41. 2	23. 9	36. 6	22. 7	. 0118	2. 76	5. 80	2. 42
583	Same plus hydrogen peroxide, steam, J-box, wash, air-dried	71	59	14. 44	42. 4	24. 0	38. 2	22. 8	. 0118	2. 64	6. 06	3. 30
584	Same as 583 but given water mangle and dry cans	71	56	9. 86	43. 3	22. 8	39. 0	22. 8	. 0114	2. 54	6. 30	3. 32
	Process No. 9:											
602	Control	64	56	0	47. 8	25. 9	47. 8	25. 9	. 0106	2. 78	5. 76	2. 14
603	Sour, wash	68	58	9. 13	46. 1	27. 1	43. 4	26. 2	. 0119	2. 83	5. 65	2. 16
604	Sour, wash, caustic saturator, steam, J-box, wash	73	56	12. 33	42. 5	24. 7	37. 3	24. 7	. 0119	2. 63	6. 08	2. 46
605	Same plus hydrogen peroxide saturator, steam, J-box, wash, air-dried	74	57	15. 03	50. 8	25. 5	43. 9	25. 0	. 0119	2. 62	6. 11	2. 85
606	Same as 605 but given water mangle and dry cans	75	55	13. 12	52. 7	24. 8	45. 0	25. 2	. 0115	2. 64	6. 06	2. 68
	Process No. 10:											
594	Control	64	56	0	46. 9	28. 0	46. 9	28. 0	. 0108	2. 72	5. 88	2. 10

595	Sour (45 minutes in acid) wash	69	59	11.96	48.4	26.4	44.9	25.1	.0118	2.82	5.67	2.16
596	Sour, wash, hydrogen peroxide saturator, steam, J-box, wash	70	58	11.72	42.2	24.6	38.6	23.8	.0115	2.74	5.84	3.92
597	Same plus second hydrogen peroxide saturator, steam, J-box, wash, air-dried	71	59	14.44	44.5	25.8	40.1	24.5	.0118	2.60	6.15	3.32
598	Same as 597 but given water mangle and dry cans	72	57	12.67	45.4	22.8	40.4	22.4	.0117	2.53	6.32	4.01
	Process No. 11:											
573	Control	64	56	0	47.0	26.1	47.0	26.1	.0106	2.75	5.83	2.26
574	Sour	68	59	10.67	45.9	26.4	43.2	25.1	.0117	2.82	5.67	2.42
575	Sour, caustic, J-box, wash, air-dry	73	58	15.36	51.7	25.0	45.3	24.1	.0118	2.84	5.63	2.98
576	Sour, caustic, J-box, chlorite and sodium hypochlorite, J-box, air-dried	73	58	15.36	45.5	23.3	39.9	22.5	.0114	2.71	5.90	12.4
577	Same as 576 but given water mangle and dry cans	72	55	9.42	45.0	19.7	40.0	20.1	.0106	2.55	6.27	11.4

[1] Fluidity at a concentration (C) of 0.5 gram of cellulose per 100 milliliters of cuprammonium hydroxide solution and the values adjusted to a velocity gradient ($\overline{G}$) of 500 reciprocal seconds (500 sec.$^{-1}$).

TABLE 12.—*Summary of analyses of print cloths which show fabric changes caused by three methods of bleaching*

Treatment	Adjusted breaking strength		Thickness	Weight	Fluidity: (C=0.5 gm. per 100 ml. $\overline{G}$=500 sec.$^{-1}$)[1]	Reflectance	
	64 warp yarns	56 filling yarns				Whiteness	Yellowness
	Pounds	*Pounds*	*Inches*	*Ounces per square yard*	*Rhes*		
Hypochlorite bleach (Processes Nos. 1–4):							
Control	47. 1	27. 4	0. 0106	2. 77	2. 11	0. 2702	0. 2505
Desized	43. 2	24. 8	. 0118	2. 70	2. 29	. 3774	. 2122
Kier boiled	42. 8	24. 5	. 0121	2. 60	2. 81	. 7023	. 0966
Bleached	42. 9	24. 8	. 0106	2. 46	5. 91	. 8092	. 0626
Peroxide kier bleach (Processes Nos. 5–7):							
Control	46. 7	27. 0	. 0106	2. 75	2. 21	. 2684	. 2509
Desized	44. 0	23. 9	. 0117	2. 78	2. 22	. 3668	. 2162
Bleached	41. 6	25. 8	. 0110	2. 55	2. 98	. 8147	. 0573
Continuous bleach (Processes Nos. 8–11):							
Control	46. 4	26. 1	. 0106	2. 75	2. 13	. 2559	. 2547
Desized	43. 8	24. 5	. 0117	2. 83	2. 30	. 3911	. 2124
First steep	37. 3	23. 5	. 0117	2. 71	2. 93	. 6082	. 1309
Bleached	41. 3	23. 5	. 0115	2. 57	3. 34	. 8115	. 0597
Average:							
Control	47. 0	26. 9	. 0106	2. 76	2. 15	. 2658	. 2517
Desized	43. 8	24. 4	. 0117	2. 76	2. 27	. 3893	. 2150
Bleached	41. 8	24. 9	. 0110	2. 52	4. 17	. 8117	. 0588

[1] Fluidity at a concentration (C) of 0.5 gram of cellulose per 100 milliliters of cuprammonium hydroxide solution and the values adjusted to a velocity gradient ($\overline{G}$) of 500 reciprocal seconds (500 sec.$^{-1}$).

Thread counts, thickness, and weight changes.—Thread counts, thickness, and weight changes were about what are generally observed in bleachery experience, revealing no unusual value. Shrinkages in area showed a range of 20 percent from highest to lowest and averaged 8.9 percent.

Breaking strengths and fluidities.—The breaking strength values after complete or partial processing of the fabrics were erratic and varied widely (table 13). The fabrics from processes Nos. 7 and 8 appear relatively stronger after bleaching, while those from process No. 5 appear much weaker. The more accurate fluidity values, however, show the fabrics from process No. 5 to have suffered less damage than those from processes Nos. 7 and 8.

TABLE 13.—*Comparison of breaking strength and fluidity changes brought about by bleaching*

Process No.	Average adjusted breaking strength $\frac{\text{Warp}+\text{filling}}{2}$			Breaking strength change from control to—		Fluidity: $\left(\begin{array}{c}C=0.5\text{ gm. per 100 milliliters}\\ \bar{G}=500\text{ sec.}^{-1}\end{array}\right)$ [2] change from control to—	
	Control [1]	Bleached, air-dried	Bleached, can dried	Bleached, air-dried	Bleached, can dried	Bleached, air-dried	Bleached, can dried
	Pounds	*Pounds*	*Pounds*	*Percent*	*Percent*	*Rhes*	*Rhes*
1	37.05	32.75	35.50	−11.60	−4.18	3.86	3.62
2	36.95	34.60	34.75	−6.36	−5.95	2.76	2.73
3	37.70	29.95	31.65	−20.69	−16.05	4.88	5.05
4	35.95	34.60	34.25	−3.76	−4.73	.97	1.05
5	36.85	29.90	30.80	−18.86	−16.42	1.00	.67
6	36.85	35.45	36.55	−3.80	−.81	1.06	.96
7	56.15	58.50	59.20	+4.19	+5.43	1.92	2.11
8	34.95	35.50	35.90	+1.57	+2.72	1.14	1.16
9	36.85	34.45	35.10	−6.51	−4.75	.71	.54
10	37.45	32.30	31.40	−13.75	−16.15	1.22	1.91
11	36.55	31.20	30.05	−14.64	−17.78	10.14	9.14

[1] Control samples contained a warp size.

[2] Fluidity at a concentration (C) of 0.5 gram of cellulose per 100 milliliters of cuprammonium hydroxide solution and the values adjusted to a velocity gradient ($\bar{G}$) of 500 reciprocal seconds (500 sec.$^{-1}$).

Breaking strengths are influenced by a number of factors, some of which are not fully understood, such as moisture content, wax removal, production of a certain harshness which causes fibers to cling together without slipping over each other, and, finally, that group of influences called "fabric assistance."

In the present investigation, the two types of fabrics used were of good commercial quality. Consequently, relationships between breaking strengths and fluidity values such as obtained by Clibbens and Ridge (*6*) on chemically degraded materials were not apparent, probably because, if present, they were masked by the other factors. While breaking strength measurements on a number of fabric samples from a single process usually indicate those damaged by faulty control, they may be very misleading when used as a measure of slight degradation in the selection of the most suitable process or procedure. In other words, breaking strengths do not always indicate the extent of fabric damage.

The fluidity (or viscosity) of solutions of cellulose in cuprammonium hydroxide, is a very sensitive measure of the extent of degradation or change in cellulose, particularly of degradation or damage in chemical processing, such as kier boiling and bleaching. Fluidities are dependent upon size of the cellulose molecules and are not affected by the many influences that affect breaking strengths.

When judged by fluidity values (table 13) all the processes except No. 11 are acceptable, and even process No. 11 falls only slightly below good bleaching standards (*10*). Of the acceptable processes, No. 3 was the most harmful. These values indicate, in contrast to the indications of other values, that the caustic soda kier boil and peroxide kier bleach was slightly better than the continuous processes, except in process No. 11, and that both of these methods are better than the caustic soda kier boil and hypochlorite bleach.

The British Fabrics Research Committee classified cottons (*9*) having a fluidity of 1 to 5 rhes as "very mildly scoured and bleached" and those having a fluidity of 5 to 10 rhes as "normally scoured and bleached." The classification was not "rigid" and the comparison of values was only "approximate."

Fluidities obtained with cuprammonium solutions of the compositions recommended by the American Chemical Society, which Conrad (*8*) used, tend to be considerably lower than those obtained with cuprammonium solutions having only 15 grams of copper per liter. Moreover, application of the Conrad velocity gradient adjustment should tend also to give lower fluidity values than those obtained by British methods. Nevertheless, the present survey indicates that the classification of the British Fabrics Research Committee, when applied to more recent bleaching processes, is still valid. Although loss in breaking strength is accompanied by increase in fluidity there appears to be no correlation between the two values (table 13).

The effects of desizing, souring, or wetting the fabrics on the breaking strength and fluidity are shown in table 14. The treatments produced losses in breaking strengths that were confirmed, in some cases, by an appreciable rise in the more sensitive fluidity values.

TABLE 14.—*Effect of souring, wetting, or desizing treatments on breaking strength and fluidity in cuprammonium hydroxide*

Process No.	Treatment	Average breaking strengths — $\frac{\text{Warp}+\text{filling}}{2}$ — Control	Treated	Change	Fluidity increase
		Pounds	*Pounds*	*Percent*	*Rhes*
1	Gray sour	37.05	34.50	−6.88	0.36
2	Malt diastase, 2 to 3 percent	36.95	30.95	−16.23	.26
3	Singe, quench, bin storage 4 hours	37.70	35.95	−4.64	--------
4	Gray sour, 95° F., cold water wash	35.95	35.20	−2.09	.39
5	Hot water dip	36.85	36.75	−.30	--------
6	Hot water dip, 18 hours storage	36.85	30.80	−16.41	.22
7	Malt diastase, 2 to 3 percent	56.15	54.00	−3.82	.41
8	Gray sour, 2.5 hours in acid	34.95	33.25	−4.86	.43
9	Acid dip, cold water dip	36.85	34.80	−5.56	.02
10	Gray sour, 0.75 hour in acid	37.45	35.00	−6.54	.06
11	Gray sour, 95° F., cold water wash	36.55	34.15	−6.57	.16

The changes produced by the malt diastase desizing treatments, processes Nos. 2 and 7, may possibly have been due to bin storage while the cloth was warm and wet, rather than to attack by the enzyme solutions. The changes produced by process No. 6, where the fabric received neither acid nor enzymic treatment but was wet with hot water and stored for 18 hours, were almost as great as those produced by process No. 2 where malt diastase was used.

The acid treatments in processes Nos. 8 and 10 differ only in the length of time the fabric remained in the acid. The fluidity values show that the fabric that remained longer in the acid (process No. 8) was considerably more degraded than the one that remained the shorter time in the acid (process No. 10). If judged by breaking strength values this damage would not be apparent.

The singe and quench in process No. 3, produced only a slight lowering of breaking strength and no increase at all in the fluidity value. Apparently the passage of the cloth through the flame was so rapid that the momentary flash of heat burned off the fuzz without producing a measurable change in the fabric.

DEGREE OF WHITENESS

The degree of whiteness produced by the processes is revealed by the reflectance measurements given in tables 15 to 17.

TABLE 15.—*Comparative whiteness of samples taken after progressive operations of the caustic soda kier boil and hypochlorite bleach*

Sample No.	Description	Whiteness	Yellowness
	Process No. 1:		
538	Control	0.2576	0.2530
539	Gray sour	.4334	.1932
540	Gray sour, kier boil	.7528	.0814
541	Same plus hypochlorite bleach, sour, air-dry	.8202	.0574
542	Same as 541 but given water mangle and dry cans	.8410	.0504
	Process No. 2:		
545	Control	.2948	.2406
546	Desize, wash	.3864	.2090
547	Desize, wash, sour, wash	.4052	.2021
548	Same plus kier boil, wash	.7410	.0837
549	Same plus hypochlorite bleach, air-dry	.8252	.0560
550	Same as 549 but given water mangle and dry cans	.8256	.0549
	Process No. 3:		
587	Control	.2583	.2578
588	Singe, quench, wash	.2937	.2412
589	Singe, quench, wash, kier boil, wash	.6230	.1246
590	Same plus hypochlorite bleach, sour, wash, air-dry	.7472	.0833
591	Same as 590 but given water mangle and dry cans	.7610	.0790
	Process No. 4 (no bleach):		
559	Control	.2401	.2549
560	Water wash	.3421	.2261
561	Water wash, sour, wash	.3321	.2310
562	Same plus kier boil, washed in kier	.6929	.1026
563	Same plus sour, sodium carbonate, air-dry	.7518	.0804
564	Same plus water mangle and dry cans	.7622	.0786

TABLE 16.—*Comparative whiteness of samples taken after progressive operations of the caustic soda kier boil, peroxide kier bleach processes*

Sample No.	Description	Whiteness	Yellowness
	Process No. 5:		
552	Control	0.2965	0.2417
553	Hot water and cold water dip	.3488	.2223
554	Hot water and cold water dip, peroxide kier bleach	.7640	.0771
555	Same plus soap wash, water wash, air-dry	.7830	.0700
556	Same as 555 but given water mangle and dry cans	.7966	.0644
	Process No. 6:		
566	Control	.2404	.2601
567	Desized	.3849	.2102
568	Desized, gray sour, wash	.3686	.2144
569	Same plus peroxide kier bleach, air-dry	.8024	.0640
570	Same as 569 but given water mangle and dry cans	.8328	.0502

TABLE 16.—*Comparative whiteness of samples taken after progressive operations of the caustic soda kierboil peroxide kier bleach processes*—Con.

Sample No.	Description	Whiteness	Yellowness
	Process No. 7:		
607	Control	.3684	.2133
613	Desize, wash	.4265	.1958
614	Desize, wash, kier boil	.6255	.1264
615	Desize, wash, kier boil, sour, wash	.7195	.0929
616	Same as 615 plus second kier boil, peroxide kier bleach, wash, air-dry	.8624	.0397
617	Same as 616 but given water mangle and dry cans	.8666	.0405

TABLE 17.—*Comparative whiteness of samples taken after progressive operations of the continuous bleaching processes*

Sample No.	Description	Whiteness	Yellowness
	Process No. 8:		
580	Control	0.2734	0.2528
581	Sour (2.5 hours in acid), wash	.3638	.2186
582	Sour, wash, caustic saturator, steam, J-box	.4670	.1772
583	Same plus hydrogen peroxide saturator, steam, J-box, wash, air-dry	.7512	.0814
584	Same as 583 but given water mangle and dry cans	.7966	.0633
	Process No. 9:		
602	Control	.2364	.2588
603	Sour, wash	.4568	.1966
604	Sour, wash, caustic saturator, steam, J-box, wash	.5582	.1483
605	Same plus hydrogen peroxide saturator, steam, J-box, wash, air-dry	.7986	.0644
606	Same as 605 but given water mangle and dry cans	.8203	.0568
	Process No. 10:		
594	Control	.2578	.2526
595	Sour (45 minutes in acid), wash	.3526	.2220
596	Sour, wash, hydrogen peroxide saturator, steam, J-box, wash	.7996	.0632
597	Same as 596 plus second hydrogen peroxide saturator, steam, J-box, wash, air-dry	.8070	.0614
598	Same as 597 but given water mangle and dry cans	.8176	.0590
	Process No. 11:		
573	Control	.2404	.2567
574	Sour	.4093	.2036
575	Sour, caustic, J-box, wash	.6880	.1024
576	Sour, caustic, J-box, chlorite plus sodium hypochlorite saturator, J-box, air-dry	.8544	.0436
577	Same as 576 but given water mangle and dry cans	.8000	.0620

These measurements, which were made on the sensitive Hunter reflectometer (*16*), show no significant differences among the three methods of bleaching. It is noticeable that process No. 4, which included no bleach at all, produced a whiteness equal to that of process No. 3, which included a hypochlorite bleach (table 15). Moreover, the whiteness produced by process No. 4 was so nearly that of process No. 5 (table 16) that sure visual detection of the difference between them would be difficult even for those accustomed to comparing whiteness differences.

Absorbability

Absorbabilities of the finished fabrics are compared in table 18.

TABLE 18.—*Comparative absorbability of samples bleached by different processes*

Process No.	Drop square		Capillary rise in 10 minutes					
			Air-dried			Can dried		
	Air-dried	Can dried	Warp	Filling	(Warp+filling)/2	Warp	Filling	(Warp+filling)/2
	Seconds	*Seconds*	*Inches*	*Inches*	*Inches*	*Inches*	*Inches*	*Inches*
1	4. 8	3. 8	3. 05	3. 05	3. 05	2. 50	2. 65	2. 58
2	4. 2	1. 4	4. 50	3. 75	4. 12	3. 75	3. 25	3. 50
3	2. 7	3. 7	3. 25	2. 75	3. 00	2. 55	2. 25	2. 40
4	2. 8	1. 6	4. 15	3. 75	3. 85	3. 75	3. 15	3. 45
5	26. 5	(1)	2. 25	1. 55	1. 90	0	0	0
6	3. 0	2. 0	4. 00	3. 50	3. 75	3. 50	3. 00	3. 25
7	2. 3	22. 3	4. 75	3. 50	4. 12	3. 25	2. 20	2. 72
8	7. 2	13. 1	3. 60	3. 25	3. 42	2. 00	. 95	1. 48
9	3. 0	2. 0	4. 25	3. 70	3. 98	3. 60	3. 00	3. 30
10	2. 6	38. 0	3. 25	2. 75	3. 00	2. 25	1. 50	1. 88
11	2. 2	2. 0	4. 00	3. 50	3. 75	3. 50	3. 00	3. 25

[1] Over 30 minutes.

Absorbability, like moisture content, is influenced by the presence of starch, soap or other residual extraneous materials, absorbed gases, the grade and staple of the fiber, count and twist of the yarns, and the temperature and relative humidity of the surrounding atmosphere. The importance of absorbability in print cloths is confined to rate of absorption (table 18), rather than total volume of liquid absorbed. Consequently, only measurements based on rate of absorption were made. The drop-square and capillary-rise methods showed very good agreement. The generally lower capillary rises in samples cut filling wise than in the warpwise strips may be attributed to differences in thread counts. In general, low absorbability occurred in samples having high alcohol solubility and wax content. There was, however, no correlation between wax content and absorbability.

When judged by absorbability, no real difference due to method of bleaching appeared.

SUMMARY AND CONCLUSIONS

Eleven bleaching processes were investigated representing three general classes of modern commercial bleaching methods: (1) caustic soda kier boil and hypochlorite bleach; (2) caustic soda kier boil and hydrogen peroxide kier bleach; and (3) continuous bleaching.

The caustic soda kier boil and hypochlorite bleach yielded fabrics with lower amounts of residual impurities (alcohol-soluble materials, wax, and ash), with higher cellulose content, than the fabrics from either of the other two processes. The continuous process showed slightly lower amounts of residual impurities, with slightly higher cellulose content, than fabrics from the caustic soda kier boil and hydrogen peroxide kier bleach.

When measured by fabric changes (breaking strengths, thicknesses, weights, and fluidities), the caustic soda kier boil and peroxide kier bleach showed slightly better results than the continuous processes using hydrogen peroxide; and both of these gave better results than the caustic soda kier boil and hypochlorite bleach. The one process using chlorite produced a slightly excessive degradation as measured by fluidities in cuprammonium hydroxide, but this observation is the result of a single processing and indicates only that careful control is imperative when chlorite is used for bleaching purposes.

As judged by reflectance measurements, no differences were revealed in the three types of processes. The slight fluctuations in the recorded values were within the limits of precision of the measurements and too small for sure visual detection.

Absorbability determinations revealed no differences between the three bleaching methods.

Desizing treatments degraded the fabrics slightly, and increased moisture contents and the apparent amounts of alcohol-soluble materials and wax in the fabrics.

High wax contents of the bleached fabrics are associated with the use of hard water and faulty washing procedures. Conversely, soft water and efficient washing procedures were responsible for removal of all but a small part of the wax content of the fabrics.

While breaking-strength measurements on fabrics from a single process usually reveal the fabrics damaged in the process, these measurements may be very misleading when used as a basis for selecting a process or procedure.

Although each of the three classes of bleaching processes had some disadvantages, all were satisfactory and produced fabrics of good commercial quality.

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