Dyeing Cotton
To Test Its Fibers

Charles F. Goldthwait

The dyes that are used to decorate cotton with different colors and with printed patterns can sometimes be employed as tests to detect chemical or physical changes in cotton or differences among cotton fibers. Thus a new dyeing test that depends upon physical differences distinguishes thin-walled from thick-walled cotton fibers. It has been used to help determine the quality of cotton at all stages from the growing boll to piece goods ready for dyeing in the finishing plant.

Because the dyed colors differ with the wall thicknesses of the fibers, they must really depend upon the mode of growth of the fibers. The cotton fiber is a tubular seed hair, a single plant cell, which grows normally by a steady secondary thickening inward of the cell wall, after a thin primary wall has grown to the full length of the fiber. The well-thickened fibers are called mature. Those with but little secondary thickening are called immature. Hence, the terms “mature” and “thick-walled” will be used interchangeably, and also “immature” and “thin-walled,” since this is the common usage, although it is not a question of maturity in a true physiological sense.

The new test is a simple, direct dyeing of any number of cotton samples at a time, with two quite different commercial dyestuffs, a red and a green, used together in the same dye bath. Certain peculiarities of the dyes and of the two types of cotton—the thick-and the thin-walled—work together to cause a pronounced differential effect.

Loose cotton samples are enclosed in gauze bags or placed between sheets of gauze with seams sewn through, so that each sample is in a separate compartment. If possible, the samples that are to be compared at one time are dyed all at once, 3 grams of each being a convenient amount. The gauze with the samples is weighed and entered into a boiling dye bath made up of 40 times the weight of cotton and containing 1.2 percent Diphenyl Fast Red 5BL Supra I (Geigy) and 2.8 percent Chlorantine Fast Green BLL (Ciba), calculated on the weight of all the cotton, samples plus gauze.

After dyeing for 15 minutes at the boil, the cotton is lifted out, and 2½ percent of its weight of pure sodium chloride is stirred into the bath. The cotton is reentered for 15 minutes, then lifted again, and a second portion of 2½ percent sodium chloride is added. The dyeing proceeds at the boil for 45 minutes, when the cotton is lifted, drained, and washed in two changes of cold distilled water in the proportion of 50 parts to 1 of cotton. After excess water is squeezed out, the dyeings are dipped into vigorously boiling water (50 to 1) for 30 seconds, with stirring. Then they are lifted, drained, and washed with cold water as before. The hot washing, really a differential stripping, is not in accordance with usual direct dyeing practice. It removes excess red dye but relatively little green, and so eliminates grayness and results in more clearly defined colors, especially in a clearer green.

After the final wringing, the dyeings are removed from the gauze bags and allowed to dry in the open air or with moderate heat. Mature cotton in its natural state will have colored to a
pronounced red. Thin-walled, or immature, cotton will be usually a distinct green.

If a better idea of the resultant color is desired than can be obtained from simple inspection of the usual dyeing, the fibers may be blended by carding. But in a simple and more effective method the samples are cut to a near powder in a Wiley mill, and the cotton is stirred into water containing a little adhesive and filtered with suction in a coarse fritted disc funnel to form a pad. The pad is removed by applying compressed air to the stem of the funnel or blowing into it. The colors of a series of the pads can be easily compared.

People have known for many years that immature cotton does not dye normally, but no dye test has been described for clearly distinguishing thin-walled fibers.

When a mill wrote to the Southern Regional Research Laboratory for help with respect to "bad-dyeing" cotton, which was causing trouble in dyeing and manufacturing and was creating factory seconds in the finished goods, I remembered a red-green dye combination that we found very sensitive to variations in cotton. Tried on "good-" and "bad-dyeing" samples from the mill, it showed a pronounced differential effect. Microscopists observed that the difference in the dyeing effect corresponded to the difference in wall thickness; the novelty of the new dye test was at once apparent, and it was soon developed to its present form.

Many other dye combinations were tried. Some showed differential effects with mature and immature cotton, but none was so satisfactory as the original red and green.

The differential dye formula has been applied in many ways to cotton—and also, incidentally, to other cellulose fibers, including rayon of different kinds, ramie, wood pulp, and cotton linters. Some of its applications are in large-scale use. The possible applications of the test to cotton begin at an early stage of growth in the boll, where it can demonstrate visually the progressive development of the lint on the seed. When the fibers have acquired their full length but have not begun the secondary thickening, they dye green in the test. After some of the thickening has taken place, they dye red. When a systematic sampling was made of unopened bolls of different ages, there was a regular increase in the proportion of red-dyeing fibers until—in a boll that opened naturally—nearly all dyed red. The test seems to indicate that thickening does not necessarily take place uniformly from end to end of the fiber. There may be green-dyeing base portions and sometimes green-dyeing tip ends, with indications that these thin-walled ends may become normally thickened later. From the test we may also be able to learn more about the way cotton grows.

The test can also be made to show that there is a submicroscopic structural change, besides the well-known deswelling and changes of shape in cotton fibers when they dry as the boll opens. If a lock of cotton taken from a boll picked before opening naturally is dyed without previous drying, the fibers will dye green, even though almost fully developed. But if a lock from the same boll is dried and then dyed, the fibers will color red, to a degree that depends upon the number of fibers with secondary thickening. Clearly, the wall thickness and all other fiber characteristics from lock to lock in a given normal cotton boll are about the same. Hence the great difference in dyeing before and after drying (from the original boll) must arise from some change in the cellulose of the cotton fiber.

The test can be applied to cotton while it is still on the seed from a boll which has opened naturally to show the relative amounts and locations of underdeveloped fibers. They are likely to be present on one end of the seed, readily seen as small tufts of green-dyed fibers, with all the rest red. Under some
conditions of growth, there may be larger tufts or some other distribution of the green-dyed fibers. The test was employed by O. J. Hunt in experimental work in the department of agronomy at Louisiana State University, where "the differential-dyeing technique gave a very accurate and consistent evaluation of fiber maturity" and "should be of practical value to the cotton breeder." Differential dyeings can also help in the selection of cotton fibers while still on the seeds for studies of fiber properties.

Similarly, the dyeings have been used extensively for the selection of fibers from ginned cotton for studying the properties of thick- and thin-walled fibers.

The exact relationship between the green-dyeing of all the fibers in the original undried condition in the boll and that of the ordinary dry, thin-walled fibers is not yet entirely clear. The differential dyeing gives the impression that all fibers grow in the green-dyeing state, which is retained until the boll opens, but that certain of the thin-walled fibers remain in that state or in some similar condition—even upon drying, when the normally thickened fibers have become red-dyeing. It seems probable that a high degree of porosity in the originally wet fibers in the boll is lost when they are dried, and not restored in the dye bath, while a corresponding porosity in the thin-walled fibers is retained, or restored by swelling in the dye bath, enabling them still to dye green.

Excessive amounts of immature fiber in cotton for manufacture may cause a number of difficulties. Thin-walled fibers, especially those with practically no secondary wall thickening, give rise to the very small tangled clumps, or neps, which cause difficulties, such as an undue number of breaks in yarn during manufacture, and defects which frequently show up as unsightly white specks in finished-colored goods. When the amounts of unrecognized immature fibers are large enough, there are frequently other difficulties in the dyeing and spinning processes. The result sometimes is a high proportion of factory seconds, because of stripes or bars across a piece of cloth meant to be uniformly dyed.

The differential dyeing gives at once a means of observing the relative amounts of immature, or green-dyed, fibers in a cotton sample. Often the nep-forming fibers can be seen in small, glazed sheets, brighter than the other immature fibers, but not dyed quite so dark a green.

The cotton trade is greeting the test as a simple and practical method for checking the character of cotton, because samples from bales containing excessive amounts of thin-walled cotton have not been readily identifiable. Several brokers are installing this qualitative test in their laboratories to help avoid shipping immature cotton to mills, which would have trouble with it. Checking every bale is especially important because even one or two bales containing excessive amounts of immature or neppy cotton can contaminate a whole blend of perhaps dozens of bales.

While some dealers in cotton are trying to forestall trouble before the cotton reaches the mill, mills themselves are making systematic differential-dyeing tests of part or all of their cotton supply. One company is making dyeings at the rate of at least 50 samples at a time, for routine checking of its cotton as an aid in selection for specific purposes before it enters production. Working on a still larger scale, another company has used differential dyeing to select bales of cotton for chambrays, where neps again may be especially objectionable as pronounced white specks of surprising prominence.

A simple procedure has been worked out to dye up to 500 samples at once, representing 500 separate bales of cotton. Each sample, bearing a part of the bale ticket for identification, is put into a small gauze bag. All samples then are put in a net laundry bag, which is placed in a small raw-stock dyeing
machine. The machine is packed with extra raw cotton to prevent channeling. Dyeing is done by the differential formula, adapted to this purpose. The samples are rinsed, whizzed, dried, and laid out for inspection. Experience tells us that too green a dye in any of the samples is evidence of a higher proportion of immature fibers and of a tendency toward an abnormal number of neps. The bales that correspond to such samples are diverted to some use where neps are not so serious.

In these last few applications, the interests of both seller and user of cotton are involved. Both could utilize the same tests, just as they employ the same techniques for estimating grade and staple. Cotton men with whom the matter has been discussed agree that a differential dyeing ought to tell things that a classer does not see readily in working with ordinary raw stock. As evidence, this whole research started from the purchasing of cotton that was about 40 percent mature and was mixed with cotton about 80 percent mature. The cottons were accepted as Middling and as Low to Strict Low Middling; although they obviously were different, they were not considered too different and went into the same blend. The result was the bad-dyeing cotton problem, which would not have arisen if the new dyeing technique had been available and had been utilized in checking the classers' samples from the bales before assigning them to mill lots.

That mill learned to reject the highly immature bales, and its production consequently increased. Such a screening can be readily made by differential dyeing; a classer who handled his ordinary white cotton in his usual way could undoubtedly learn to tell much more about the cotton if he had differential dyeings to go along with his regular samples. Not only could he reject the worst bales; if he were experienced in observing the dyed cottons, he could draw conclusions regarding their character as well as the properties covered in the usual estimates of grade and staple.

The test promises to be useful in another way in cotton manufacture. If samples of the processed cotton and of corresponding waste from different steps in manufacturing are dyed, it is possible to follow the blending-in of the immature fibers and check final uniformity. It is apparently possible also to determine something of the nature of the waste. In a few trials on samples from the experimental manufacturing unit of the Southern Laboratory, there was no observable tendency to remove in carding any greater proportion of immature fibers than was left in the carded cotton. In combing, however, a somewhat larger proportion of immature cotton was removed. The observations indicate what can be done. They are not to be taken as necessarily representative for all cottons or for all conditions of manufacture. However, the method seems worth trying for comparing the running of different cotton and possibly for comparing different machine settings to obtain more efficient production and better quality.

Other applications to yarn and piece goods may be to correct specific faults and difficulties—or they may be embodied in routine testing. The test serves well to show up mixed yarn, including that in cotton piece goods—in one case, streaks in a warp pile fabric. Similarly, a pronounced change in shade may occur at a seam joining two different lots as they go through a dyeing process. The differential dyeing can be used to predict whether the cottons in such lots are likely to dye differently and to cause serious trouble.

Although some applications of differential dyeing amount to estimates of maturity, there is need for a quantitative technique, preferably by a simple dyeing and inspection of the resulting color.

The over-all color obtained on almost any cotton sample will enable it to be placed correctly in a series of dyed samples in the order of percentage of maturity. So far, visual obser-
DYEING COTTON TO TEST ITS FIBERS

vation has seemed adequate if the fibers are cut up and made into pads as I have suggested, but the color may also be estimated by optical measurements.

While the results have already been suitable for mill use, numerous difficulties have been encountered in the development of the test as a quantitative method. Recognized difficulties lie in the reproduction of dyed shades with such a two-color combination (especially when different cottons are being dyed nearly every time) and in the influence of exposure of open bolls in the field to weather, which can alter the ultimate dyed shades. Factors of unknown nature also have caused anomalous results from time to time, as compared with the results of conventional maturity determinations.

However, the point should be made that the results from dyeings need not check with maturity determinations, and the colors obtained may eventually prove to be even more useful than maturity figures. In support of this possibility, the dyeing is made on a larger sample and includes the full lengths of all the fibers; it is not merely an estimate of the relative wall thicknesses at their midportions.

While the real need is for the rapid method based on a simple observation or measurement of color, it has been necessary to make many regular maturity counts, and the occasion was taken to try the method first used by O. J. Hunt—counting the numbers of red and green cross sections in differentially dyed samples.

The method, as it was adapted at the Southern Laboratory for ordinary samples of cotton, consists in dyeing, sampling for the microscope, cross-sectioning, and counting the red and green fibers in a total of 1,200. Although it is a microscopical method not too greatly different from those ordinarily used (which employ either swelling by caustic soda or viewing by polarized light), operators who have used all three prefer the colored cross-section method. One reason is that there are no questionable fibers, all being easily placed as green or red. A possible additional advantage is that it is based on cross sections of natural fibers just as they are, rather than on fibers distorted by a high degree of swelling, or on somewhat uncertain optical effects.

While differential dyeing generally follows wall thickness or maturity, as commonly understood, it is almost certainly not actually due to wall thickness or to maturity in a more strictly physiological sense. The same general effects are obtainable with cotton fibers, which are seed hairs, with ramie bast fibers, and with man-made rayon. Hence it seems clear that the differential dyeing depends upon fine-structural features of cellulose which are not peculiar to cotton. This view is supported by the observations on dyeing cotton wet from the boll as already described. The properties of the two dyes used also enter into the picture. Whatever the complete explanation, most of the applications to cotton—whether in growing, trading, or manufacture—are associated with effects having to do with so-called maturity or immaturity.

In sum, the detection and estimation of immaturity in cotton are of special importance at all stages—from the growing cotton, where immaturity originates, to the finished goods, where it may lower the value of the product.

Charles F. Goldthwait was graduated in chemistry from Worcester Polytechnic Institute. For several years he was in charge of a laboratory that served a group of textile mills. He gained wide experience with woolen, worsted, and union goods, the processes of mercerizing, bleaching, and dyeing, the manufacture of textiles from seed flax, and rayon and silk. Later he joined the Mellon Institute of Industrial Research, and worked there for nearly 15 years on research on the processing of cotton. Since 1941 he has been in charge of a varied program of research on cotton in the Southern Regional Research Laboratory.