

A Method of Differentiating  
Fibers in Textile Fabrics

by O. Lecomte

1910

Senior Thesis

The amidogen group in silk and wool is diazotized by nitrous acid, and the resulting compound gives characteristic colorations in alkaline solution with various phenols. Wool, again, contains sulphur in addition to the amidogen group, and this will react with lead to form a black sulphide, which will mask the diazo-coloration. Vegetable fibers give neither reaction, and can thus be readily distinguished from animal fibers.

The reagents required are: (1) Dilute nitric acid, 100 gr. per liter. (2) Sodium ~~plumbite and sodium~~ nitrite solution, fifty gr per liter. A solution of sodium plumbite and sodium naphtholate, prepared by dissolving 50 grams of sodium hydroxide in 500 c.c. of water, and adding little by little 25 gr of lead subacetate solution (French Pharmacopœia) in 300 c.c. of water. When the liquid is clear 5 grams of B-naphthol are added, and the solution made up to a litre. (4) A solution of sodium plumbite and sodium resorcinate containing 25 grams of lead subacetate, 50 grams of sodium hydroxide, and 2 grams of resorcinal in a litre. (5) Dilute hydrochloric acid, 5 grams per litre.

One square decimeter of the bleached fabric is introduced into 30 c.c. of the dilute <sup>nitric</sup> acid, and when thoroughly impregnated is treated little by little during 3 minutes with 30 c.c. of the sodium nitrite solution. Throughout this addition and for some time after, the material is constantly stirred and pressed with a rod against the side of the beaker. After ten minutes it is thoroughly washed for two minutes and cut into equal portions. One of these is treated with 40 c.c. of reagent 3, and the other with 40 c.c. of reagent 4, at a temperature not exceeding 20C., the reactions being complete in an hour. The fragments are then washed for 15 minutes in running water, then immersed for 5 min. in dilute HCL, and again washed for at least an hour in running water, after which they are pressed between filter paper and dried in the dark.

When examined under the microscope, the portion treated with reagent

ent3 will show any silk fibers of a rose color, wool fibers black, and vegetable fibers colorless. In the other portion the silk fibers will be orange wool fibers black, and vegetable fibers colorless. The proportion of the different fibers can be readily found by counting in the usual way.

A COLOR REACTION OF OLEIC ACID AND ITS USE IN DISTINGUISHING VEGETABLE FROM ANIMAL FIBERS. A. MANEA.

When a substance such as cellulose, starch, celluloid, dextrin, or a sugar, is dissolved in strong sulphuric acid, and the solution treated with a few drops of oleic acid or an oil containing oleic acid, and then with water, added drop by drop with constant shaking, there is produced an intense red coloration, which changes to violet on the further addition of water. If the latter be added without shaking, a very characteristic <sup>colored</sup> ring is obtained. Animal fibers do not give this coloration, and the test affords a rapid means of distinguishing between, e.g., true and artificial silks. If the fibers have been sized it is necessary to boil them with water, and to dry them ~~dry~~ again before applying the test. The presence of dye-stuffs does not affect the production of the coloration. Certain fractions of crude petroleum, especially those distilling about 250°C., give the same colorations as oleic acid in this reaction.

## TESTING THE FASTNESS OF COLORS.

Matthew's  
Laboratory Manual! Page 292-299.

(1) Fastness to Light:—A sample of the dyed wool is placed in a suitable frame in such a manner that only a part is exposed. The frame is then placed in such a position that it receives as strong sunlight as possible, but is shielded from exposure to the atmosphere by glass. A window with southern exposure is a good location in which to hang the frame containing the samples. At the end of one week's exposure the samples are examined and note made of those which show any appreciable fading; these are to be classified as not fast. At the end of the second week another examination is made and those samples noted which show an appreciable fading; these are to be classified as fairly fast. At the end of four weeks the samples are once more examined and the colors fading in this period are noted and classified as fast. The samples which show no fading at the end of four weeks are classified as very fast.

(2) Fastness to Washing:— This test is to represent the fastness of a dye to washing or scouring with soap and water. Scour the sample for 10 minutes in a miniature scouring bath (about 50 cc.) containing 5 grams of soap per litre at a temperature of 140°F. Squeeze, wash off in fresh water, and dry. Note if the dye tints the soap solution, and if it tints either the white wool or the white cotton.

1. - Fast; does not tint the soap liquor, nor either of the white yarns.
2. - Fairly fast; tints the soap liquor, but not the white yarns.
3. - Not fast; tints either of the white yarns; the soap liquor may or may not be tinted.

(3) Fastness to Fulling:— Treat with a solution containing 10 grams of soap and 2 grams of soda ash per litre at 140°F. Soak the sample in this solution and rub between two pieces of board until the wool yarns are well felted together. Then wash in fresh water, and dry. Note if the color has lost in intensity or if it has bled into either the white wool or cotton. In such case the dye cannot be considered fast to fulling. According to the degree of bleeding, the color may be classed as not fast or as fairly fast. If the dye neither loses in color nor bleeds, it may be classed as fast.

(4) Fastness to Rubbing:— This is also termed "crocking", and refers to whether or not the dye will mechanically rub off, and thus stain white or other colors with which it may come in contact. Heavy shades are more apt to rub than light shades. As a rule, the acid and substantive dyes on wool do not rub; the basic dyes frequently show this defect; heavy shades of mordant (or pigment dyes in general) will frequently rub off to some extent, whereas lighter shades do not. Heavy shades of indigo (a pigment dye), for instance, rub off considerably. The test for fastness to rubbing is easily and simply carried out by rubbing a portion of the dyed sample on a piece of white calico, and noting if a stain is left.

(5) Fastness to Water:— Boil with water for 1 hour. If the dye does not bleed at all in boiling water it may be classed as fast; if it bleeds slightly in boiling water, but not in cold water, it is fairly fast; and if it bleeds in the cold water test, it is not fast.



(6) Fastness to Weather:—The best and most practical method of applying this test is to expose a sample of the dyed material to the action of the weather for two weeks or more. But the results may be approximately represented by the following test. (a) Steep a sample of the dyed yarn in a solution containing 2 parts of hydrogen peroxide (10-volume strength) and 10 parts of water for 1 hour. Dry and compare with the original sample. (b) Repeat the test using a hydrogen peroxide solution of 10-volume strength undiluted with water; steep for 1 hour, and on drying compare with the original sample. If no alternation in the color is appreciable after test (b), the dye may be classed as fast; if test (b) shows an alteration in the color, but not test (a), the dye may be classed as fairly fast; if test (a) shows an appreciable alteration, the dye is not fast. By combining this test with the one to sunlight, a fair idea of the fastness of the dye to weather exposure may be gained.

(7) Fastness to Acids and Carbonizing:—In many cases dyed woolen materials are treated with moderately strong solutions of acids and dried in order to decompose any particles of vegetable matter which may be present; this process is known as carbonizing. To test the fastness of a dyestuff to this operation, proceed as follows: Immerse a sample of the dyed yarn in a solution of sulphidic acid of 4°Tw. at 175°F. for half an hour; squeeze and without washing dry in a hot air flue. Then wash out and neutralize the acid in a bath containing about 1 gram of soda ash to 100 cc. of water; finally rinse well, and dry. Compare with the original color and note if the carbonizing process has altered the shade in any manner. According to the extent of change in the shade, classify as not fast, fairly fast, and fast. Test in this manner each of the ten dyed samples.

(8) Fastness to Perspiration:—This is required of all dyed clothing material that is worn next the skin; also of material used for making horse-blankets, etc. The most reliable test is to wear a sample of the dyed wool in such a manner as to expose it to the action of perspiration. This action, however, may be well represented by the following test: Plait a sample of the dyed yarn with white woolen and cotton yarns, and immerse for 1 hour in a solution of acetic acid of 4°Tw. 1.02 sp. gr. at 100°F. Squeeze, and dry without washing in the air. Note if the color has suffered any alteration in shade or if it has stained either of the white yarns. According to the extent of change or staining classify as not fast, fairly fast, or fast.

(9) Fastness to Alkali:—In order to remove the fatty matters from woolen goods a washing with dilute soda ash solution is frequently given. This is especially true of material which is fulled. To discover if the color will withstand such a treatment, a test is made as follows: A sample of the dyed yarn is plaited with white wool and white cotton, and steeped for one hour in a solution of soda ash 3°Tw. at 120°F., then washed in fresh water and dried. Note if the color suffers any alteration, or if either of the white yarns is stained. Accordingly to the extent of change in color or staining the dye is to be classified as not fast, fairly fast, or fast.

(10) Fastness to Street Dust:- Dyed clothing materials such as ladies' dress goods and gentlemen's suitings have to withstand the action of street dust, mud, etc. This action is best represented by a test with lime as follows: Spot a sample of the dyed yarn with a solution containing 20 grams quicklime and 10 cc. ammonia per litre. Allow this to dry on the material, and then brush off. Note if the color has suffered any alteration.

(11) Fastness to Ironing:- Woolen material employed in the manufacture of suitings, etc., requires to be hot pressed or ironed. To discover if a dyestuff will withstand such a treatment, test as follows: (a) Moisten a sample of the dyed yarn and press with a hot iron till dry. Note if the color undergoes any alteration on cooling. (b) Moisten a sample of the dyed yarn and cover with a piece of white muslin, then press with a hot iron until dry. Note if the color suffers any alteration or if it stains the white muslin. If no change takes place under (a) class the dye as fast; if it changes under (a) but not under (b), or stains the white slightly without any other perceptible change, class as fairly fast; if the color is altered by both (a) and (b) class as not fast.

Mathew's "Laboratory Manual". pp. 310 - 313

Experiment 168. To Determine the Amount of Wool and Cotton in a Fabric:— A weighed portion of the sample is boiled for 10 min. in a 5 per cent. solution of caustic potash, then washed well first with fresh water and afterwards with water slightly acidulated with acetic acid to remove all trace of alkali. The residue is dried and weighed. As the cotton present will suffer a slight loss in the process, it is customary to add 5 per cent. of its weight to the cotton, and to subtract this amount from that of the wool.

Experiment 169. Analysis of Fabric Containing Silk and Cotton:—(a) Nickel Hydrate Method. A weighed portion of the fabric (about 5 grams) is steeped for 5 minutes in a cold solution of nickel hydrate in ammonia; then heated almost to boiling for 5 minutes. This treatment should dissolve the silk completely. The residue of cotton is thoroughly washed, dried, and weighed.

(b) Zinc Chloride Method:— A weighed portion of the sample is boiled for 2 minutes in a solution of basic zinc chloride of 1.72 sp. gr. The residue of cotton is thoroughly washed first with dilute hydrochloric acid, and then with water, and then dried and weighed. This treatment dissolves the silk without materially affecting the cotton.

Experiment 170. Analysis of Fabric Containing Wool and Silk:— A weighed portion of the sample is steeped for 2 min. in concentrated hydrochloric acid at 120°F. This will dissolve the silk without materially affecting the wool. Wash the residue of wool, dry, and reweigh.

Experiment 171. Analysis of Fabric Containing Wool, Silk and Cotton:— A weighed portion of the sample is treated for 10 min. with a cold solution of nickel hydrate in ammonia (see above). This will dissolve any silk present. Wash well, dry, and reweigh. The loss in weight represents silk. The residue is next boiled for 10 minutes in a 5 per cent. solution of caustic potash. This will dissolve any wool present. Wash well, dry, and reweigh. The loss in weight represents wool, while the residue consists of cotton (see Reagent 11 for correction to apply to weight of cotton).

Experiment 172. Distinction between True Silk and Artificial Silk:— To estimate the amount of artificial silk present in a mixed fabric, a weighed portion of the sample is treated at the ordinary temperature for 20 minutes with an alkaline solution of copper sulphate. This will completely dissolve the natural silk, leaving the artificial fibre as a residue. The latter is thoroughly washed, dried, and reweighed.

Experiment 173. To Distinguish between Cotton and Linen:-

(a) Steep the sample containing these two fibres for 2 minutes in concentrated sulphuric acid; wash well with water, gently rub with the fingers, and finally steep in dilute ammonia; then squeeze and dry. The cotton fibres will be converted into a jelly-like mass by the action of the acid, and is more or less completely removed by the rubbing and washing. The linen remains but little altered. By weighing the sample before and after the treatment an approximate idea of the amounts of cotton and linen present may be obtained. (b) Steep the sample to be tested in olive oil; then press between filter paper to remove the excess of oil. The linen fibres will become gelatinous in appearance and translucent, whereas the cotton remains unaltered. When placed on a dark background the linen fibres will now appear dark and the cotton fibres light. (c) Steep the sample to be tested in an alcoholic solution of fuchsin, and then in a strong solution of caustic soda; finally rinse in water. The linen fibres will become rose-colored, while the cotton is colored much lighter and most of the color is removed by the rinsing. None of these tests are very satisfactory when the linen has been bleached for then its cellulose is practically identical with that of cotton. The most satisfactory means of qualitatively distinguishing linen from cotton is by a microscopic examination, as these fibres exhibit very different microscopic properties (see Matthew's Textile Fibres).

Experiment 174. To Distinguish between True Silk and Tussah Silk:- Tussah silk (and the wild silks in general) may be distinguished from the true silk by the following reactions:

(a) Tussah silk is only partially dissolved by cold concentrated hydrochloric acid (sp. gr. 1.16), even on standing for 48 hours; whereas true silk dissolves almost instantly.

(b) Tussah silk requires a comparatively long time to dissolve in the solution of basic zinc chloride, mentioned in Exp. 168; whereas true silk dissolves quite readily.

(c) True silk dissolves completely in a semi-saturated solution of chromic acid when boiled for 1 minute; whereas tussah silk remains unaltered after boiling for 2 or 3 minutes in this solution.

(d) To estimate the amount of tussah silk in a fabric, weigh off a portion of the sample and steep for 10 minutes in cold concentrated hydrochloric acid; wash the residue thoroughly, dry, and reweigh. The loss in weight represents the true silk while the residue consists of tussah silk.