

CHEMICAL SECTION.

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SOME RECENT DEVELOPMENTS IN TEXTILE PROCESSES.

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The cry of the civilized world is, and has always been, for novelty. In the multitude of mechanical and industrial pursuits which engross the attention of the busy worker in laboratory and shop, wonder succeeds wonder; the world, astonished perhaps for a moment, looks on with languid interest, and then demands further proofs of the fact that we are but on the threshold of industrial development.

The fickleness of fashion, the demands of an ever-active competition, and the various problems presented by the inexorable laws of supply and demand, have ever been a powerful stimulus to the textile worker, as well as to his co-laborers in other fields of industry.

A few developments of comparatively recent origin seem to promise new avenues for the skill of the textile worker, and it is my purpose to present to you in a few brief, practical notes, a number of new applications of chemistry to textile processes which are engrossing the attention of American and European mills and dyehouses.

On account of its high luster, great strength and elasticity, and its affinity for nearly all artificial and natural coloring matters, the fiber excreted by the many species of silk worm has always held the highest place in the estimation of textile-manufacturing nations.

The great demand for silk for ornamental and useful purposes, and its high intrinsic value, have been the incentives for many efforts towards its artificial production.

While nothing has yet been brought forward which has been capable of displacing silk in the textile industry, interest-

ing progress has been made in the art of imparting a silk luster and appearance to other and cheaper fibers.

As early as 1844 John Mercer, a cotton printer, of Lancashire, England, discovered that, when subjected to a strong solution of caustic soda or caustic potash, cotton fibers became greatly contracted in length, as well as more dense, and to a high degree capable of being directly dyed with basis dye-stuffs.

He made this discovery the basis of broad patents, which were issued to him in England and in continental countries in 1850, and, to quote the original patent application, his claims were as follows:

“The invention consists in subjecting vegetable fabrics and fibrous materials, cotton, flax, etc., either in the raw or manufactured state, to the action of caustic soda or caustic potash, dilute sulphuric acid, or chloride of zinc, of a strength and temperature sufficient to produce the new effects, and to give the new properties to them which I have hereafter described.

MERCER'S INVENTION DESCRIBED.

“The mode I adopt of carrying into operation my invention to cloth, made wholly or partially from any vegetable fibers, bleached, is as follows: I pass the cloth through a padding machine charged with caustic soda, or caustic potash, at 60° or 70° Twaddell's hydrometer, at the common temperature, at say 60° F. or under, and, without drying the cloth, wash it in water, then pass through dilute sulphuric acid and wash again, or I run the cloth over and under a series of rollers in a cistern with caustic soda or caustic potash at from 40° to 50° of Twaddell's hydrometer, at the common temperature of the atmosphere, the last two rollers being set so as to squeeze the excess of potash back into the cistern; the cloth then passes over and under rollers, placed in a series of cisterns charged at the commencement of the operation with water only, so that, at the last cistern, the alkali has been nearly all washed out of the cloth; when the cloth has either gone through the padding machine or through the cisterns above described, I wash the cloth in water, pass it through dilute sulphuric acid, and wash it again in water.

“When I adapt the invention to gray or unbleached cloth, made from the fibrous material before mentioned, I first boil or steep the cloth in water, so as to have it thoroughly wet, and remove most of the water by the squeezers or hydro-extractor, and then pass the cloth through the soda or potash solution, etc., and proceed as before described.

MERCER'S INVENTION APPLIED TO WARPS.

“I apply my invention in the same way to warps, either bleached or unbleached. By this process I produce on cotton and other vegetable fabrics and fibers effects somewhat analogous to that which is produced on woolen by the process of fulling or milling. It will have acquired greater strength and firmness, each fiber requiring greater force to break it. It will also have become heavier than it was before it was acted on by the alkali, if in both cases it be weighed at the temperature of 60° F. or under. It will have acquired greatly augmented and improved powers of receiving colors in printing and dyeing.

EFFECTS OF THE APPLICATION.

“The effects of the application of my invention to the vegetable fiber in any of its various stages before it is manufactured into cloth will be readily understood by reference to its effects upon cloth composed of such fibers.

“Secondly, I employ sulphuric acid diluted to 105° Twaddell's hydrometer, and at 60° F. or under. I use this acid mixture instead of caustic potash or soda, and operate in all respects the same as when I use soda or potash, except the last souring, which is here unnecessary.

“Thirdly, when I employ solution of chloride of zinc, instead of soda or potash, I use the solution at 145° F., and operate in all respects the same as when I use soda or potash.

“When I operate on mixed fabrics, partly of vegetable and partly of silk, woolen, or other animal fibers, such as delaines or leans, etc., I prefer the strength of the alkali not to be above 40° Twaddell's hydrometer and the heat not above 50° F., lest the animal fibers should be injured.

CONCLUDING REMARKS.

“I may, in conclusion, remark that the description of the apparatus or machinery and the strength and temperature of the soda or potash, sulphuric acid or chloride of zinc solution, may be varied to a considerable extent, and will produce proportionate effects without at all deviating from my invention. For instance, caustic potash or soda may be used even as low as 20° Twaddell’s hydrometer, and still give improved properties to cotton, etc., in receiving colors in printing and dyeing, particularly if the heat be low, for the lower the temperature the more effectively the soda or potash acts on the fibrous material above described. I, therefore, do not confine myself to any particular strength or temperature of the substances I employ, but the particular strength, heat, and processes here described are what I have found the best, and what I prefer.

“And I claim as of my invention the subjection of cotton, linen and other vegetable fibrous material, either in the fiber or any stage of its manufacture, either alone or mixed with silk, woolen or other animal fibrous material to the action of caustic soda or caustic potash, dilute sulphuric acid, or solution of chloride of zinc of a temperature and strength sufficient to produce the new effects, and to give to them the new properties above described, either by padding, printing or steeping, immersion or any other mode of handling.”

Notwithstanding the great expectations aroused among cotton manufacturers by Mercer’s patents, so-called mercerized cotton has, until recently, assumed little importance because the contraction of the fiber in all directions made cotton cloth so treated much more expensive, and the advantage gained never appealed to the handler as being sufficiently strong to compensate for the increased cost of production.

Within the last two years, however, an accidental discovery in connection with mercerizing cotton has opened a new field of usefulness for this process, and the silk-lustering of cotton yarns or cloth by means of solutions of caustic alkalies or similar reagents, presents many possibilities.

A textile chemist in Germany, whose name at this writing escapes my memory, was experimenting with a fabric com-

posed of equal parts of silk and cotton with a view to the acquisition of some means by which piece goods of this description might be colored evenly in one bath.

In one experiment he treated his cotton yarns with a strong solution of caustic soda while holding same in a stretched condition, and, to his great surprise and delight, noted the fact that the cotton fiber had assumed a silky luster and appearance, and could be dyed in very fast and bright shades without losing its fine silk-like appearance.

As a result of this happy accident, manufacturers in all parts of the world have been experimenting on similar lines, and, through the courtesy of the J. R. Montgomery Company, of Windsor Locks, Conn., which is very successfully manufacturing these silk-lustered cotton yarns on a large scale, I am enabled to present for your examination some very handsome samples of this yarn, which is easily dyed and may readily be woven into durable and handsome fabrics.

To obtain this silk luster and finish, the best method to be pursued is as follows: Yarns, warps or piece goods in a stretched condition are immersed in a solution of sodium hydrate (caustic soda) for about fifteen minutes, strength of bath to be about 52° Twaddell.

The cotton goods thus treated are then passed through squeeze rolls, or are hydro-extracted, to remove excess of alkali, and are then thoroughly rinsed with clear water.

A second bath of dilute sulphuric acid suffices to neutralize all remaining alkali, when a thorough final rinsing leaves the yarn in condition for either dyeing or bleaching.

The character of yarns employed for this purpose has a marked influence on the luster and finish obtained, as soft-twisted and double yarns made from long staple Egyptian or Sea Island cotton give decidedly the best results. Hard-twisted yarns and single yarns of ordinary cotton acquire luster and a greater affinity for dyestuffs, but they are inferior in appearance to those made of soft-twisted double yarns from Egyptian or Sea Island cotton.

As a result of the uniform contraction of the fiber, due to this process, these yarns have a tensile strength of from 25 to

40 per cent. greater than before treatment, and this property adds greatly to the advantage to be derived from the use of such material in the manufacture of articles for ordinary wear.

The silk touch and feel or "scoop," as it is called, may be obtained on mercerized yarns by treating them after dyeing or bleaching with alternate baths of olive-oil soap and calcium chloride, as follows:

The dyed or bleached yarns (or cloth) are first passed through a bath made up of one pound neutral olive-oil soap dissolved in twenty-five gallons of water. The yarns are then hydro-extracted and passed through a 1 per-cent. solution of calcium chloride, and afterwards washed and steamed lightly.

This precipitation of a lime soap (principally calcium oleate) on the fiber imparts the peculiar silky feeling so much desired in the manufacture of dress goods, hosiery, underwear, etc.

Where this scoop is not of particular benefit, it is always well to soap the dyed or bleached yarns or goods with a good neutral olive-oil soap, which adds softness and improves the luster.

To insure even lustering I have always found that it is better to use a weaker solution of alkali and longer immersion therein, rather than a stronger caustic bath and a shorter treatment.

A 52° Twaddell solution of caustic soda is as strong as necessary, and the average time of immersion should not be over fifteen minutes.

Stretching and twisting on suitable machines after the mercerizing, also tend to heighten luster, and are recommended to all handlers of this material.

Since the first notes on this method of silk-lustering appeared, many other reagents have been recommended in the place of the caustic alkalies, but few if any of them appear to present any practical advantage.

One method, patented in Germany, calls for the use of a mercerizing bath made up of 30 per cent. of sodium or potassium sulphide, to which is added 10 per cent. of any of the following fat solvents (which float on top of the alkaline sulphide solution, so that the yarns or cloth pass first through the sol-

vent and then through the sulphide), methyl or ethyl alcohol, benzol, aniline oil, paraffine oil, petroleum or turpentine.

As the yarns or goods treated with caustic soda or potash are, as a rule, boiled out and freed from impurity before entering the bath, this method, which I have carefully tried, costs more than the other, and does not seem to give any better results.

Another application of this mercerizing process to textile fabrics is mentioned in a late number of the *Textile Manufacturer*, of Manchester, England, from which I quote:

GLOSS ON COTTON AND LINEN GOODS.

“Among the very recent processes for obtaining a silk-like gloss effect on fibers and fabrics is one devised by the Farbwerke, of Hoechst-am-Main, and described in the *Textile Manufacturer*, of Manchester, England.

“The object of this is to produce durable effects on both cotton and linen stuffs. To do this they employ the modified mercerizing system of Thomas & Prevost, by applying appropriate resisting styles capable of neutralizing the mercerizing effects of soda lye upon the fabric in the state of tension. By these means they claim to obtain a new, valuable and technical effect. It is known that it has been impossible hitherto to produce damask-like gloss effects of perfect durability by means of the dyeing or printing processes. The methods employed until now were, as a rule, limited to printing with oxide of zinc, barium sulphate, etc., combined with a fixing agent (albumen, caseine, etc.), on tissues of sateen-like weaving and gloss dressing in order to thus neutralize the sateen gloss, and of the tissue by means of this locally-fixed white color. While the gloss produced by this sateen weaving cannot be compared with real silk gloss and with that obtained on cotton by Thomas & Prevost’s system, and must be regarded merely as an effect of weaving and a particular method of dressing, it is to be observed that the silk gloss of mercerized cotton in a state of tension is obtained by a chemical and physical change of the cotton fiber.

“It is a fact that the mercerizing of the cotton in a state of

tension causes the lumen of the fiber to shrink, to become transparent, and to undergo great changes on its surface which produce this lasting silk-like gloss. Also, from a chemical point of view, the fiber undergoes an advantageous change, and it is generally known that, since Mercer's experiments, the affinity of cotton fiber towards mordants and dyestuffs is considerably increased by the influence of strong soda lyes and agents acting in a similar manner (concentrated sulphuric acid, solution of chloride of zinc). It is, therefore, possible to produce this mercerizing effect on fabrics locally, under conditions as specified by Thomas & Prevost (tension), either by means of printing with concentrated soda lye or by protection of the fiber against the mercerizing effect of the soda lye, a durable and damask-like effect being thus obtained. The substances suitable for the resisting style are easily coagulable organic bodies, such as albumen, caseine, etc., as well as salts, acids or oxides, which partly have a neutralizing effect, or provide the fiber with a protecting film of an oxide.

"The resisting action of these bodies against soda lye is not new. With the help of these bodies the most varied white and colored gimping effects were obtained, while the object of the present process is to avoid the shrinking of the tissue and the silk gloss thus caused.

"In the description of a German patent particular stress is laid upon the circumstance that the printed resisting style—albumen, caseine, etc., or mixtures of gum with acetates of aluminium or chromium—becomes insoluble by previously steaming, and thus forms an inseparable compound with the fiber. In order to obtain the intended effect, the applied resisting styles need not be steamed, and the possibility thus created of effecting a subsequent removal of the albumen, or the encrusting protecting substances, is in many cases a great advantage. As the silk-like gloss produced on the tissues in form of patterns, according to this method, is very durable, such cloths may be afterwards dyed, printed, steamed and washed without danger to the gloss. It is likewise possible to produce the most varied printing effects with durable silk gloss by the addition of mordants or dyestuffs to the resisting styles or to the mer-

cerizing soda lye. The printing of the resisting styles and the mercerizing may be done separately or in succession on the printing machine; in the latter case the printed cloth, on leaving the roller, is stretched by means of an appropriate stretching apparatus. The washing in a stretched condition is continued until the tension of the inner fiber has ceased.

“The following is an example of an appropriate printing color:

“Mercerizing color for direct printing, 70 grams British gum and 930 grams soda lye (40° B.), or 100 grams wheat starch, 200 grams water, and 2,000 grams soda lye (40° B.).

“*Resisting Color.*—Albumen solution, 10°, 700 grams, and dilute solution of gum tragacanth 300 grams.

“We might add further that the microscopical appearance of cotton mercerized by the process of Thomas & Prevost differs from that of ordinary cotton in a striking and typical manner. The mercerized fibers mounted in water generally appear stretched and smooth, only showing the twisted appearance in places, or not at all. Those fibers which possess the well-known twisted appearance have a large lumen and are little changed. The fibers from the outside portion of the yarn show, in consequence of the greater tension, longitudinal folds, while those from the inside frequently show transverse crushed folds.

“The surface of the fibers often shows an intermittent double stripe. The cuticular layer is generally completely demolished. The lumen of the fibers in places is greatly enlarged; in others it appears as a dark line, while in others it has entirely vanished. The enlarged portions of the lumen are frequently filled with a granular mass. In polarized light the fibers behave just like ordinary cotton.”

The extensive use of mohair and lustrous wools in the manufacture of braid yarns and dress goods, etc., has led to many experiments with ordinary wools to secure silky appearance and luster, and while a perfectly satisfactory process has not yet been obtained, some very interesting results of a more or less practical value have been arrived at through the use of various oxidizing agents, most prominent of which are chlorine and bromine in dilute solutions.

The combination of fatty-acid precipitation with energetic oxidation by means of sodium or calcium hypochlorite is the basis of the following formulæ, which have given me good silky luster, but which have a more or less tendering action on the fiber.

For light shades on 20 pounds of knitting yarn, take the soluble part of 3 pounds of calcium hypochlorite (bleaching powder) dissolved in 150 gallons of water, with from 3 to 4 pounds of HCl. Work for from 30 to 45 minutes at 140° to 158° Fahr.

For dark shades use, for same quantity of yarn and same amount of water, 6 pounds hypochlorite of calcium and 3 pounds HCl, working for 45 minutes at 122° Fahr.

For use on either dark or light shades take 100 gallons sodium hypochlorite liquor, testing ½° B., with 6 pounds HCl, and treat for 30 minutes at 122° Fahr.

Another method, perhaps better on account of lessened tendency to yellow the fiber, is to treat for 30 minutes at 86° to 95° F. with a bromine solution of from 5 to 8 per cent. strength; but, while this method has a better effect on the strength of the fiber than chlorine, the luster is less brilliant.

In all cases much care is necessary that the operation be not prolonged beyond the specified time, that the temperature be not permitted to rise too high, and that the yarns be turned constantly so as to insure uniform oxidation.

As wool fiber is always more or less discolored by this treatment, it is necessary to bleach before dyeing, aqueous sulphurous acid being best adapted for this purpose. Silk scroop is imparted by charging either before or after dyeing with a solution made up of 120 grains of olive-oil soap per gallon of water, to which is added 50 cubic centimeters of 10 per cent. H₂SO₄.

Temperature of this broken soap bath should be about 122° Fahr.

Another beautiful product of chemical industry is the artificial silk made under the patents of Lehner of Zurich and the Compté De Chardonnet, of Bordeaux, which, while not yet of demonstrated practical value on account of high cost of pro-

duction compared to silk and various mechanical defects, yet furnish a valuable starting point for what certainly should be a flourishing industry when these defects are overcome.

If cotton be carefully cleansed and passed through a cold bath of strong nitric acid, it is converted into dinitrocellulose without becoming particularly tender or inflammable.

Pyroxylin or gun cotton of the composition $C_{12}H_{14}(NO_2)_6O_{10}$ belongs to the same group of nitrated cellulose compounds, and by means of less concentrated acid we obtain a less nitrated trinitrocellulose known as soluble pyroxylin, which is soluble in alcohol and ether, forming a heavy syrupy fluid known as collodion.

But it is the tetra-nitro cellulose of the same group which forms the basis of the interesting results obtained at different times by Lehner, Chardonnet and others in the artificial silk industry before mentioned.

To produce the remarkable fiber, of which I am able to show typical samples, the pyroxylin, in the form of the tetra-nitrocellulose, is dissolved in a mixture of 38 parts ether and 42 parts alcohol to form a 6.5 per cent. pyroxylin solution.

This solution is introduced into a tinned copper receptacle, from which it is forced by continuous air pressure through a vertical glass tube, ending in a fine capillary orifice, and surrounded by a second glass tube, through which there is a steady flow of cold water.

As soon as the nitrocellulose solution comes in contact with the water it solidifies and may be drawn from the tube as a continuous thread, which, however, is highly inflammable, and must be de-nitrated before it can be safely used.

This is accomplished by first treating with dilute nitric acid, and afterwards with a solution of ammonium phosphate.

The artificial silk thus obtained may now be colored in the same way as ordinary silk, care being taken to avoid boiling, which greatly injures its strength, the temperature of the dye bath being best fixed at or below 180° F.

The greatest faults of this cellulose silk lie in its lack of elasticity and its great loss of strength on becoming wet, to which its hygroscopic nature makes it peculiarly liable.

However, I am informed that the use of 15 per cent. of formaldehyde, acetic aldehyde, paraldehyde or other aldehyde derivatives in the solvent used for the pyroxylin will yield a fiber free from these defects, and that the product of the Chardonnet factories is now so treated.

In this connection it might be added that formaldehyde presents to the textile chemist many very useful characteristics, which are being taken advantage of in several processes now in use, and which we will briefly note.

The product formaldehyde was first obtained by Von Hoffman, in 1867, by passing the vapor of methyl alcohol, mixed with air, over finely-divided Pt heated to redness.

It is also formed in small quantity, together with marsh gas (CH_4) and formic acid $\text{H}_2\text{CO}_2 = \text{HCOOH}$, by the action of silent electric discharges on a mixture of H and CO_2 . It may be abundantly obtained at a low cost by the incomplete oxidation of methyl alcohol in a combustion furnace or lamp suitably constructed, and has the formula $\text{CH}_2\text{O} = \text{HCOH}$.

Commercial formaldehyde or formalin is a solution of the gas in water, and has come into great prominence through its wide application as a disinfectant. To the textile chemist the property possessed by formaldehyde of rendering gelatine and other bodies insoluble in water suggests many useful applications in dyeing, color printing and finishing.

Besides its use in adding to the strength and durability of cellulose-artificial silk, it may be used in obtaining a silky fiber of considerable strength by the following method:

Gelatine is dissolved in water and a syrupy solution obtained. Then, by a device somewhat similar to that used in the Chardonnet and Lehner processes, it is drawn out into a thread, which is immediately passed into a 4 per-cent. solution of formaldehyde, which renders the fiber insoluble and capable of being woven or knitted in with other fibers.

In this method the dyestuffs would, however, be better added to the gelatine solution, as, after fixation by formaldehyde, the silky fiber obtained would be very difficultly penetrated by the coloring matter, and unsatisfactory results would follow.

A solution of gelatine, prepared in a dark place, with from $\frac{1}{2}$ to 1 per cent. of bichromate of potash, has also been suggested as a method of obtaining a textile fiber from gelatine, as, owing to the well-known sensitiveness of chrome gelatine to light, it becomes insoluble on exposure, and this derivative has one advantage in being capable of absorbing dyestuffs after fixation. For light colors it is unsuitable, as it always has a dark appearance.

Whether this fiber is destined to hold any practical position in the textile arts is a matter of mere conjecture, as its production so far has not passed beyond the purely experimental stage.

In sizing the backs of velvets and other fabrics, a gelatine solution, colored with any desired dyestuff, is brushed evenly over the surface to be sized, and then the material is either sprayed or brushed over with a 2 per-cent. formaldehyde solution, giving a very permanent and useful size.

In color printing the dyestuffs are added to a gelatine solution of the desired density, and, after the colors are steamed on, the cloth is passed through a 2 per-cent. formaldehyde solution, with the result of fixing the color on the fiber in combination with insoluble gelatine. This reaction has suggested several uses for formaldehyde in printing cotton goods.

In waterproofing it would furnish a uniform coating, which could be cleansed without injury, and capable of being handled very inexpensively. For coating silk or other varieties of fishing lines, it furnishes an enamel which is more permanent and waterproof than any which have so far been used, is entirely without deleterious effect on the fiber, and is already being employed for that purpose, as the samples I have here will testify.

In Lyons, formaldehyde is being extensively experimented with in the manufacture of so-called souple silks, in which the sericine or silk gum is merely softened sufficiently to enable its being handled, and is not boiled off, as is the case with tram and organzine silks.

Formaldehyde has the property of fixing or rendering insoluble this sericine, and is used after the soupling and dyeing

to prevent loss of weight in the different processes to which it is subjected.

There are doubtless other applications in which this insoluble gelatine would be very useful, and another fact, that regarding its property of converting fuchsine and other aniline reds into blues and violets, might not be without hint of possibilities to the colorist.

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