Machinery and Appliances.

*APPARATUS FOR THE MANUFACTURE OF ARTIFICIAL SILK FROM LIQUIDS.**

By M. Hilaire de Chardonnet.

This paper is intended to describe the apparatus for the manufacture of artificial silk. In order to understand properly the function of each part, it is necessary to remember that artificial silk consists essentially of nitrated cellulose in a state of solution. This solution projected into another liquid, which coagulates it, is formed instantly into a thread which can be gathered and treated like a thread from a cocoon. In order to lessen the high combustibility of this substance, a part of its nitric acid is taken from it by a carefully managed process of separation, and it is made at the time to absorb different saline and colouring matters. The operator takes some of the purified cellulose obtained from chemical pastes of wood or straw, cotton, rags, filter-papers, hemp, ramie, etc. This is then nitrated by well-known methods to render it soluble in a mixture of alcohol and ether.

A more or less concentrated collodion is formed by dissolving pyroxyle in a mixture containing 40 per cent. of ether and 60 per cent. of alcohol in volume. The pyroxyle is first dissolved in equal parts of alcohol and ether; and then the excess of alcohol is added. This solution, well filtered, is introduced into a machine for spinning liquids, to the description of which we now pass.

The accompanying illustrations represent the mechanical appliances used in the process. Fig. 1 gives a front view of the machine; Figs. 2, 3, and 4 enlargements of details; Fig. 5 end elevation; fig. 6, cross section with attachments; figs. 7 and 8, enlargements of details.

The collodion solution, well filtered, is introduced into a closed receiver, not shewn in the illustrations, in which an air-pump maintains a pressure of from 10 to 12 atmospheres. This receiver, which is tinned inside, communicates by a tube provided with a cock with the tube A, which bears the spinners and extends all the length of the machine. This tube A, shewn on a larger scale in front and section in Figs. 2 and 3, is composed of three compartments: the central one B, which receives the solution, and two lateral ones C and C¹, which serve to maintain a circulation of water round the central tube B. Each spinner is composed of a tube *a*, to which is attached a capillary tube *b*, and the lower orifice of which communicates with the cylinder *c* screwed in the tube A; the mouth of this cylinder opens in the solution. A cut socket D keeps each spinner on the corresponding cylinder, and makes the joint tight by means of two washers of leather or some other substance, which press between them the lower part of the tube *a*.

The flow of the liquid is regulated by means of the pin *f*, borne by the bar *h*, which can be screwed more or less into the cylinder *j* placed at the bottom of the tube A, in order to check or facilitate the passage of the liquid on its way into the spinner. Round the extremity of each of the spinners *a* is found a tube-envelope *k*, supported by the plate *l*, which extends the length of the machine. The tube *k* brings cold water to the extremity of the spinner; this cold water comes from the tube D, which distributes it to all the tube-envelopes by means of the taps E. These taps serve to regulate the flow of the cold water in which the thread is immersed on leaving the tubes *a*; it is got rid of by the gutter F¹, which runs along the length of the machine, and forms the rim of the plane of the plate *l*, which is inclined transversely.

When the machine is not working, the tubes A and D, which bring the water and the liquid, are kept closed, and the orifice of the spinners is stopped by a drop of mineral oil, which prevents all contact of the air with the liquid or the water. When starting, it is sufficient to re-establish the pressure in the tubes A and D; the liquid bladder of collodion is solidified by its passage into the water, and instantly forms a thread, which, drawn by the water, bends round the tubes *k*, where it is gathered up by means of a special nipper, shewn in Figures 3, 4, 6. Each of these nippers consists of two plates *m*, which are suitably curved so as to embrace the corresponding tube-envelope, and present an oblique part which, gliding over the tube-envelope, forces the pincers to open when they arrive against the tube *k*. All these pincers *m* are carried by a bar *n*, which is connected at each of its extremities with a square *o*, articulated with the two arms *p*, *p*¹, (Fig. 6), one of which receives an oscillatory movement, drawing on the other by means of the square *o*. These arms are moved in the following manner: their axes bear on the outside of the box F, a crank *q* is then put in motion by the connecting rod *r*, which is moved by the toothed wheel r_1, which is actuated by the pinion *s* fixed on the shaft of the motor pulley *l*. The shaft of the pinions S and *t* of the wheels r^1 extends the length of the machine.

Under the influence of the oscillatory movement of the arms *p* and p_1, the two nipper-bearing squares occupy either the position indicated on the plan, or that indicated by the dots.

At the commencement of the work, the material having overflowed above the tube-envelopes *k*, the springs forming the nippers stick to the growing threads when the nippers rise; these threads are lifted on horizontal guides *u* and on other guides in the form of forks, which collect them by groups in order to bear them to the bobbins K. These grouped threads adhere by capillarity and form a raw material with several fibres. When once the work of spinning has commenced there is no solution of continuity between the orifice of the spinner and the bobbin; the material proceeding from the spinner is constantly wound on to the bobbin which corresponds to it. If the thread breaks the new end is taken up by the nipper, as was said above. At the upper extremity of their course the nippers rub against a revolving brush, which cleans them. This brush consists of a cylinder on which are placed the plates *v*, which scrape the nippers and take away all the superfluous matter attaching to

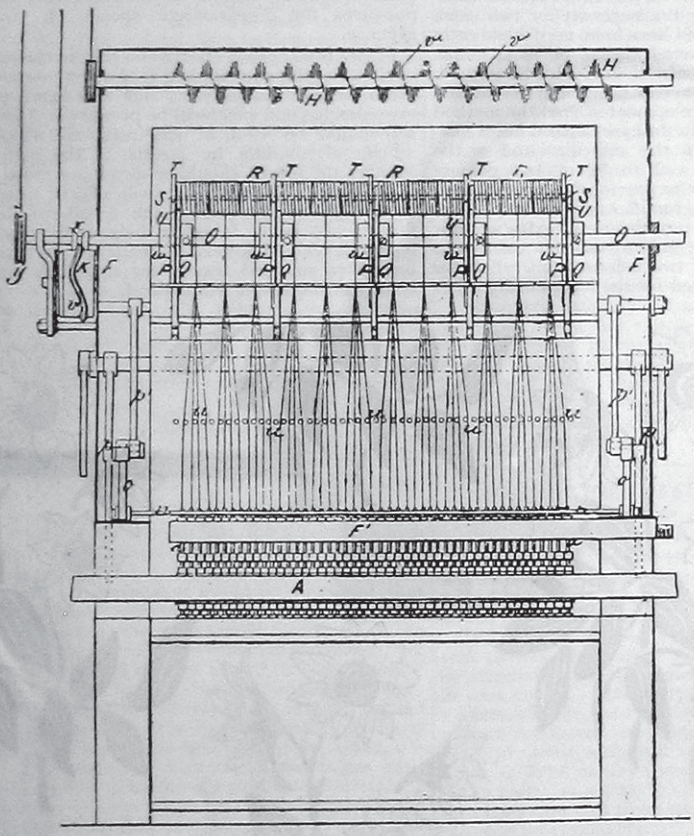

FIG. 1.

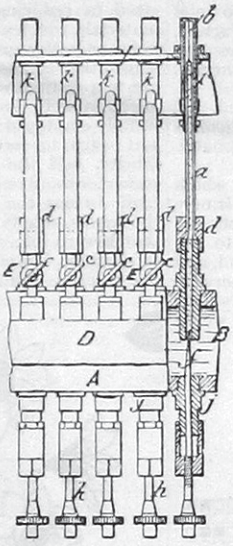

FIG. 2.

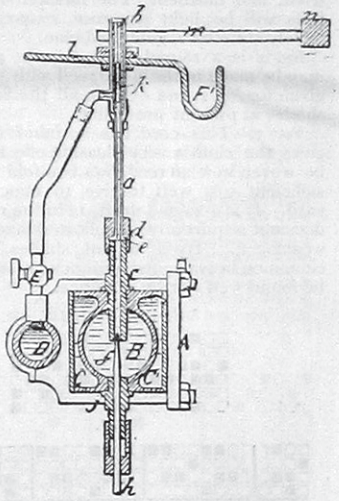

FIG. 3.

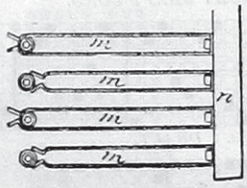

FIG. 4.

* From *L'Industrie Textile*, to which we are also indebted for the illustrations.

them. The plates v of the brush H are so arranged as to form a helical curve. The nippers are alternately long and short, as is shewn in Figure 4, in order that they may not act all together on the tube envelope k, so as to avoid or at least to lessen the shock caused at the moment of their opening. Through their opening alternately the effect is more regular.

A current of air heated to about 50° enters by the lower part of the box F and rises to the upper part, charged with vapours of ether and alcohol. To recover the vapours of the solvent it is preferable to make the hot air pass on, leaving the machine through three chambers, chilled by a current of water, two of which are shewn in Figure 6.

The first chamber f^1 contains water saturated with an excess of carbonate of potassium. The water carried away is condensed by dissolving the excess of carbonate of potassium. The alcohol and ether (in part) are also condensed; but, being insoluble in the aqueous liquid, they form on its surface a distinct layer, which is made to flow out by a gauge-tap g^1 according as the products accumulate; another tap below, the ventilator or the blowing machine, as was said above. It is advisable to have three charges of sulphuric acid, of which two are in use. When the acid of the first is saturated, the air proceeding from the carbonate is sent into the second and third condensers, whilst the first is empty and charged afresh. Thus a constant movement is established between the three chambers. In order that the successive turns of the thread on the bobbins may not overlap each other and cross each other angularly the bobbins receive a horizontal movement or traverse. This movement is produced by a cam K formed by a drum, which presents an undulating edge jutting out v, which during the rotation of the cam carries to the right and left the sort of friction-roller x fixed on to the axis of the bobbins, whilst the latter turn by means of the grooved pulley y in relation with a suitable transmission. This friction-roller is formed of two spherical knobs of steel, which can be brought into proximity according to the measure of wear of the helicoidal tooth, which is cut so as to fill in all positions the interval of the two knobs.

The threads might be received on reels, but this method would involve the inconvenience of dismounting the shaft bearing the reels whenever it was desired to take away the skeins, these latter having to undergo a special reeling as in ordinary silk-works.

The properties of artificial silk admit of a better process of reception of it on the bobbins themselves. These bobbins are mounted on special clamps, where they are maintained by springs.

The shaft O, which has a continuous rotary movement and at the same time a rectilinear one, and alternates according to its axis, bears a series of clamps mounted on it and on the discs Q. The bobbins R are strung on spindles S, which are kept on the clamps P by plated springs T; and each of the spindles S, which serve to them as axes of rotation, presents a small friction-roller U, which, being in contact with the circumference of the disc Q, which corresponds to it, causes the spindle to turn, and consequently the group of bobbins mounted on this spindle, which has for this purpose a square transversal section.

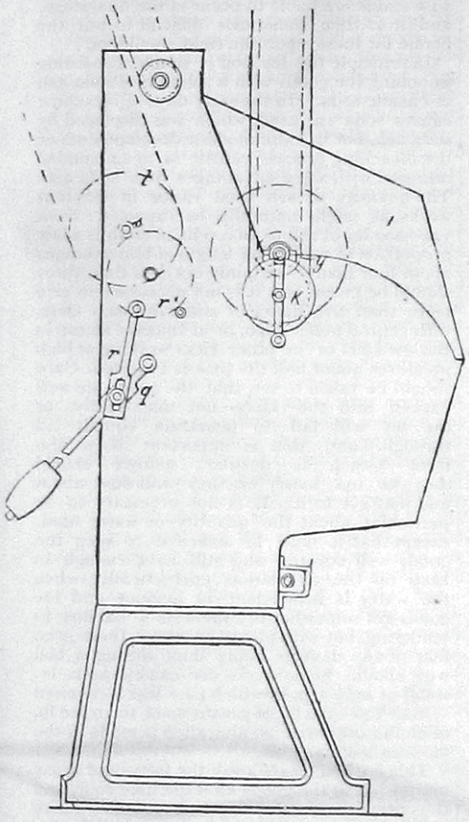

FIG. 5.—END ELEVATION.

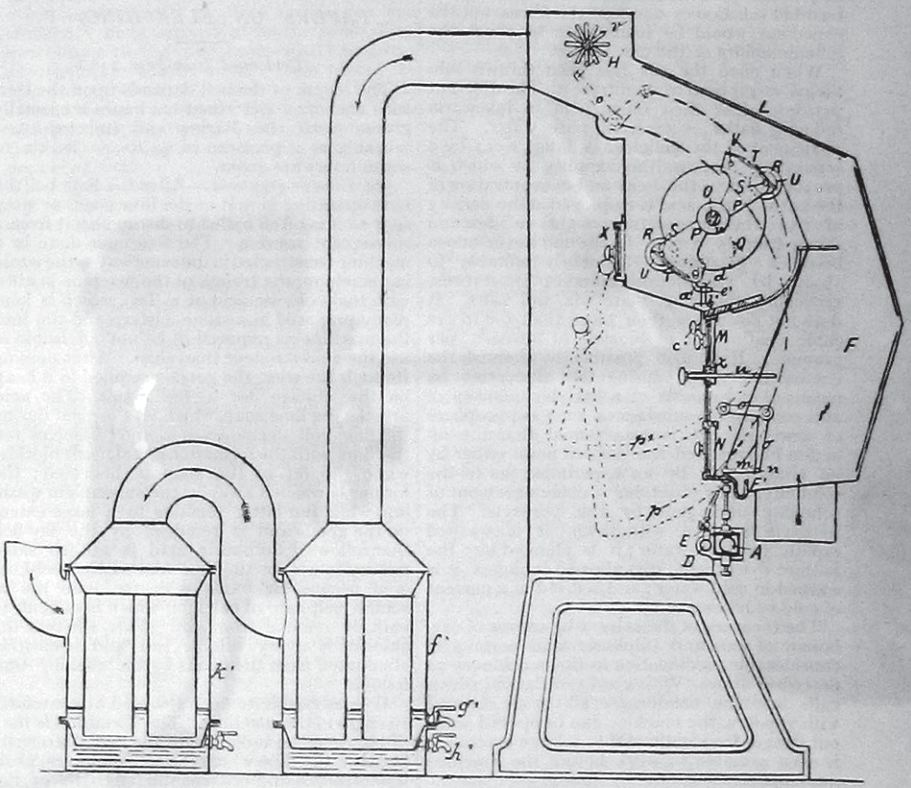

FIG. 6.—CROSS SECTION; WITH ATTACHMENTS.

h_1 serves to make the condensed water flow and to maintain the level of the carbonated solution. The dissolved carbonate can be restored by evaporation and serve indefinitely. Almost all the alcohol and a part of the ether therefore remain in the first chamber. A simple passage in contact with carbonate of potassium, followed, if necessary, by a distillation, restores the alcohol and ether to a condition of fitness for subsequent operations; a considerable portion of the ether is carried away by the current of air.

The second and third chambers k^1, of which only one is represented, are charged with concentrated sulphuric acid, which detains the ether and the remains of the alcohol. When the sulphuric acid of the second chamber has absorbed about four times its volume of ether it is withdrawn; it is diluted with water, which separates about three-fourths of the condensed ether, and distillation separates the remaining fourth. This ether, after a passage over carbonate of potassium, and if necessary a distillation, is ready to be used again. The air thus dried is sent back to the spinning machines by

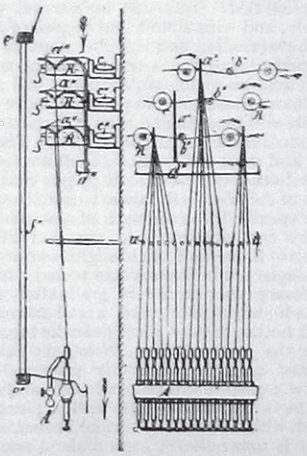

FIG. 7. FIG. 8.

The clamps P are all connected with each other by bars V, which permit us to turn the whole system, to withdraw the full bobbins, and replace them by empty ones. It is possible to avoid turning the two systems of bobbins at the same time by giving them a little play to the eye n of the clamps P. The weight of the system admits of the application of the friction-rollers U of the upper bobbins on to the discs Q, and to disengage the friction-rollers of the lower bobbins in such a way that these latter do not turn.

In order to facilitate the removal of the loaded bobbins and the replacing of them by empty ones, a series of small valves X is arranged permitting of passing the hand into the cage of the machine.

To ensure the position of the system during its working there is put at each extremity a fork a^1 which embraces the extremity of the bars V which is in the lower part; this fork permits the lateral movement of the system which bears the bobbins, but prevents it from being drawn in the direction of the rotation of the shaft o. The forks a^1 are mounted on rods

b^1, which can be made to ascend or descend from the interior by means of the buttons c^1, and their position can be regulated by the screw d^1, which presses on the tail of the forks a^1 so as to place the upper bobbin in the most suitable position for the enrolling of the threads of silk.

The cage of the apparatus is glazed in front and behind. It presents a lid L jointed in the upper part.

The water which moistens the spinners is charged with alcohol and ether, which must not be lost. Thus it is always the same water constantly returned by a pump, which serves indefinitely.

On the side where the worker, charged with the superintendence of the machine, is stationed, are two rows of slide-valves to permit of cleaning the beaks or horizontal guides u.

It is known that fibres contract a certain amount of adhesion during drying; this permits of reeling and throwing them like ordinary silk. If the adhesion is not found sufficient, some adhesive may be introduced into the water which moistens the fibres. It would also be possible to introduce in this way anti-combustible substances amongst the fibres, but the expedient would be inadequate to check the inflammability of the new textile.

When once the silk has been thrown into skeins we proceed to de-nitrate it. The different pyroxyles lose their nitric acid in lukewarm reducing baths or even in pure water. The nitric acid of the cellulose is taken away by a separating process, the rapidity of which is proportioned to the heat and concentration of the bath. Nitric acid is employed at the density of 1·32; the temperature ought to descend slowly from 35 to 25°. In the end the cellulose becomes gelatinous, eminently suitable to absorb by endosmosis different substances, especially colouring materials and salts. It does not disengage then more than 100 to 110 cubic centimetres of bioxyde of nitrogen per gramme. It is also possible to diminish the combustibility by causing the absorption by means of endosmosis of a variable quantity of anti-combustible substances, such as phosphate of ammonia. It is ascertained that the reaction has reached the desired point either by an analysis, or by an experiment as to the solubility, or by observing a commencement of solubility undergone by the pyroxyle. The pyroxyle is then withdrawn; it is washed rapidly in cold water; it is plunged into the bath of dye or salt; it is allowed to digest; it is washed in cold water; and is dried in a current of cold or lukewarm air.

The recovery of the solvent by means of carbonate of soda and sulphuric acid permits of considerable modification in the machinery as described above. With good ventilation, which calls into the condensers all the air charged with vapours, the machine can be opened without danger frequently and to a large extent; it is even possible to work before the machine quite open.

The mechanism can then be reduced to the following arrangements, represented by Fig. 7, which shews a cross section of the machine, and Fig. 8, which shews an elevation. These figures exhibit the arrangement of the guides, the pulleys, and the yarn whilst working.

The beaks rise freely above the matter-tube A, and support only their tube of water. After having traversed the comb, formed by the fixed horizontal guides u, the threads are placed with the hand on the guides a^{11} and on the bobbins R. These bobbins, which are ordinary throwing-spools, are strung on the spindles S like throwing spindles, but turning horizontally and perpendicularly to the wall of the works by means of pulleys connected with spindles turning in a small metal frame fixed by itself in the wall, under the action of a friction-strap as in ordinary throwing sheds. These pulleys c^1 and their spools turn all in the same direction, and the adhesion of the pulley is secured by small return-pulleys b^{11} similar to the pulleys c^{11}, but not bearing spindles. The guides in a^{11} are thick wire or twisted copper, and all fixed on a cross-bar d^{11} moved horizontally with a movement of come-and-go perpendicularly to the wall. This movement is obtained either by means of a pulley, as is done in the large machine, or by means of one of the numerous methods employed in engineering and in spinning.

The heated dry air arrives from the condensers from above and is exhaled below, drawing with it the vapours heavy with ether and alcohol.

The front of the machine can be closed at will by means of the glass-case f^{11} running in the grooves e^{11}.

Two spools of three are at work. As soon as a spool is full the worker causes the bundle of yarn to pass on the empty turning spool, and replaces the full spool by an empty one, which awaits its turn.

The spools hold on to their spindles simply by friction, as in ordinary throwing factories.

Bleaching, Dyeing, Printing, etc.

PAPERS ON BLEACHING.—V.

(*Continued from page* 136.)

The length of the boil depends upon the kier: with the open kier about ten hours are usually given; with the Barlow and Injector kiers, working at a pressure of 40 to 50 lb., six to seven hours are given.

5th. Lime or grey sour.—After the lime boil the next operation is that of the lime sour, or grey sour as it is often called to distinguish it from a subsequent souring. The souring is done in a machine constructed in the same way as the washing machine; the trough of the machine is filled with hydrochloric acid at 2° Tw., which is kept ready prepared in a stone cistern and run into the machine as required (it is not advisable to use the acid stronger than this). After passing through the sour, the goods are piled in a heap on the stillage for a few hours. The acid attacks the lime soap, which was formed during the lime boil, decomposes it, and dissolves out the lime with the formation of calcium chloride, while the fat of the soap is liberated; the former is washed away in the subsequent washing, while the latter remains to a large extent on the goods and is removed by the ley boil that follows. Sulphuric acid is not so satisfactory to use for the lime sour as hydrochloric acid, because it forms with the lime the insoluble sulphate of calcium, which is difficult to entirely remove from the goods, whereas the chloride is a very soluble salt and is entirely eliminated from the goods by the washing that follows.

It is advisable to keep the acid at a uniform strength in the machine. The Twaddell is here of no use as an indicator of the actual strength, because the lime, which the acid dissolves, while it neutralises and reduces the strength of the acid, actually raises the Twaddell, under which circumstances the only safe method is a chemical test. This can be carried out very simply, and with a sufficient degree of accuracy by the workmen, and if it be done at regular intervals during the souring, and the supply of fresh acid be regulated, the sour will be kept at a more uniform strength, and more uniform results will be obtained than if the souring were done in a more empirical fashion. The test is best and most easily done as follows:—Prepare a solution of the Newcastle 77 per cent. caustic soda of 20° Tw. (By always using the make of soda specified, the strength of various batches of the test solution will vary but little from time to time, and such slight variations may be neglected in these works tests.) It is now necessary to prepare a graduated measure, for which purpose take a tall narrow white glass bottle, make a mark near the top, and fill it with the test solution. Now take exactly five ounces' measure of freshly prepared sour of 2° Tw.; pour into a jar, and add carefully some of the soda test solution until a piece of cloth dyed with turmeric is turned brown, when the acid is neutralised; now make a mark on the bottle of soda to shew how much has been used. (In all subsequent tests of the sour 5 oz. should always take the same quantity of the soda solution; if it takes less it is too weak; if more it is too strong; the remedy in each case is obvious.) It is worth while to graduate the test bottle for 1°, 3°, 4°, and 5°, as well as for 2° Tw.

The goods should not be left too long piled up after souring, as they may become dry either entirely or in parts. In any case, as the goods dry the acid becomes concentrated, attacks them, and makes them tender, which is not at all desirable. Therefore, if it is not convenient to wash them for some time after souring, they should be moistened with water from time to time, but it is best to wash them off at once, whereby they are made ready for the next operation.

6th. Ley boil.— This is perhaps the most important operation in the whole process of bleaching, and unless it be well done a thorough bleach cannot be obtained. It is more especially important that the ley boil be thorough and the goods well bottomed when they are going to be printed in the so-called madder style with alizarine colours, or otherwise stains are liable to occur in the final stage, and it is then sometimes difficult to put the blame for these upon the right shoulders.

In principle the ley boil is simple, consisting in boiling the goods with a solution of soda ash or caustic soda. In the early days of bleaching caustic soda was used, which was displaced by soda ash, but in more modern developments of the bleaching process caustic is again coming into use, with some advantages over soda ash. The quantity of ash used varies in different works, as might naturally be expected: from 170—200 lb. of ash to 10,000 lb. of cloth is a fair proportion to use. The length of boil averages about four hours—certainly not less than three should be given, and it is not necessary to give more than five hours in either ordinary kiers, with central puffer pipe, or in Injector kiers; in Barlow kiers or in other kiers working at high pressures about half the time is required. Care should be taken to see that the goods are well packed into the kiers—not too tightly, or the ley will fail to penetrate equally all through, and this is important if a uniform bleach is desired; neither should they be too loose, or they will float about and may get torn. It is not necessary to be particular about the quantity of water used, except that it must be sufficient to keep the goods well covered and still have enough to keep up the circulation energetically; when the water is insufficient in amount and the goods get somewhat dry there is a liability to tendering, but with plenty of water there is no fear of any damage being done during a boil with alkali. Some works use caustic soda instead of soda ash, in which case less is required —from 120—150 lb. of caustic soda to 10,000 lb. of cloth; otherwise no alteration is made in the mode of boiling.

This ley boil clears away the fatty and waxy matter left in the goods after the lime sour, and thus prepares the way for the next boil. There is no advantage in this preliminary boil in the use of caustic soda, the cheaper soda ash being just as effective. It is found best to run the ley into the kiers cold, and to boil up; some bleachers are in the habit of "sweetening" the goods by first running in sufficient of the ley to cover them, leaving for a few minutes for the goods to become impregnated; next to run off and blow steam into the kier, then, when the goods have got warm, to run in the ley and boil up. After the boil the goods are washed, when they are ready for the next boil with resin and soda.

(To be continued.)

A YELLOW colouring matter is obtained by treating polychromine with ammonia. It forms a light brown coloured powder, easily soluble in water, and barely soluble in alcohol, and dyes unmordanted cotton from a salt bath.

LOGWOOD EXTRACT of good quality will contain 50 to 52 per cent. of colouring matter, 6 to 8 per cent. of organic bodies, about ½ per cent. of insoluble matter, about 0·12 of mineral matter, and about 40 to 42 per cent. of water.