DYEING, BLEACHING, FINISHING, Etc.

THE MAKING OF ARTIFICIAL DYE-STUFFS.

The majority of the artificial dyes are made from coal tar products. The object of this article is to show how a dye is made from the crude coal tar.

Coal tar is a side product, obtained by the distillation of coal, in the manufacture of illuminating gas. It is a black viscous liquid, composed of many compounds which are separated from one another, principally by distillation. The coal tar is introduced into a still, and distilled until a thick residue is obtained, this is generally accomplished between a temperature of 530° to 570° F. Sometimes it is carried to a higher temperature, all depending on the quality of residue wanted. This residue is pitch.

The oils which distil over are redistilled and are separated into different fractions. The first fraction includes all the oil that distils over up to 340° F., and is known as light oil. The second fraction includes the oils that distil between 340 to 400° F., and is known as heavy oil. The third fraction is that which distils between 400 to 430° F., and consists mainly of napthalene or coal tar camphor. The residue contains *anthracene oil* and many other complex products.

The light oil is now treated with concentrated sulphuric acid, to remove tarry matter which it still contains. The sulphuric acid is then drawn off and the oil washed with water; the water is allowed to settle and is then drawn off. The oil is now washed with a weak solution of soda (NaOH) to neutralize any acid that may remain; the solution after settling is again drawn off. The last traces of soda are washed away with water. The washed oil now undergoes fractional distillation, in a column still, (a column still is used for careful distillation). The product that distils between the temperature of 174 to 178° F., known as *benzene* or *benzol* is caught in one fraction, and the product that distils between the temperatures of 233 to 237° F., known as *toluene* or *toluol* is caught in another fraction. It is from benzene and toluene that most of our artificial dyes are indirectly made, and it is necessary that the benzene and toluene be made very pure by repeated fractional distillation. Benzene and toluene are clear colorless highly refractive liquids. They have a characteristic odor which is not at all unpleasant; they are so similar in appearance that the best way to distinguish them is by their boiling point; benzene boils at 176° F. and toluene at 233° F.

The benzene is now treated with a mixture of strong sulphuric and nitric acid and nitro benzene results. This operation is carried out in cast iron vessels, three to four feet in diameter, and three feet deep. The mixture of nitric and sulphuric acid is slowly added to the benzene, to prevent the action from becoming too violent and for the same reason in the early part of the operation these vessels are cooled by a water jacket through which a current of cold water is constantly passing. Later on, as the reaction becomes more quiet, the cold water is shut off and hot water is passed through the jacket.

The nitro benzene floats on the top of the mixed acids which are now drawn off and the nitro benzene is carefully washed with water, and dilute soda, to remove and neutralize the acid which remains; it is then washed with water until all traces of the alkali are removed. Toluol is nitrated in exactly the same way as benzol, the resulting product being nitro toluol. Nitro benzene is a clear yellow liquid with an odor resembling that of bitter almonds. It is commonly known as oil of myrbane and is used to a large extent for scenting cheap toilet soaps.

Although there is only one nitro benzol, there are three nitro toluols, which have exactly the same amount of chemical elements, carbon, hydrogen, oxygen and nitrogen, in them, but they are arranged in a different manner. The most important of the three nitro toluols is *ortho* nitro toluol. All three are formed in the nitration of toluene, but the *ortho* is formed in the greatest quantity, the manufacturers making it a point to obtain those conditions which will give the greatest yield of *ortho* nitro toluene. The nitro toluene obtained is similar in appearance to nitro benzene. One of the nitro toluenes obtained, the *para*, is a solid, but this dissolves in the ortho nitro toluene which is a liquid.

The nitro benzol and toluols are now converted to their respective amido compounds, aniline and toluidine. This is done by treating the nitro product with ground iron filings and hydrochloric acid. This operation is carried on in a cast iron clyinder fitted with a stirring apparatus. When the reaction is complete, lime is added and the aniline is distilled with steam.

The heavy oil from coal tar contains a substance very important in the manufacture of artificial dyes, this substance is *phenol* or carbolic acid. This is extracted from the heavy oil by treating it with a 10% solution of caustic soda and agitating the mixture in an iron tank for a number of hours. After a while the mixture is allowed to settle, the soda solution containing the phenol separates from the oil, and is drawn off. This soda solution is now treated with dilute or weak acid, to neutralize the soda, and carbolic acid, or phenol is set free. This separates out as a black oily liquid which can be easily separated from the water solution. This oily liquid is crude phenol.

This crude phenol is then refined first, by fractional distillation, and finally by crystallization which is accomplished by placing the distilled carbolic acid in cans which in turn are placed in a cold brine solution for 18-30 hrs.; by the end of that time the pure carbolic acid crystallizes to a solid mass, and is separated from its impurities which are liquids, by draining them off. Carbolic acid or phenol is too well known to require a description.

The third fraction, that which distils between the temperatures 400 to 430° F. contains napthalene. This third fraction is cooled to about 50 or 60° F., and when the napthalene crystallizes out. The oil is drained off and the napthalene that remains is then subjected to hydraulic pressure to eliminate as much oil as possible. It is then washed with sulphuric acid

loop *b* in Fig. 21 corresponding to loop *a* in Fig. 18 for the next stitch. In the illustrations, loop *b* is represented shaded, for clearness, and the small arrows indicate the direction the loops travel.

The needle is now ready to receive yarn again and make another stitch, the completed stitch and the last formed loop being pressed down in position by the cloth wheel. When the needle comes round again to the feed carrier, another loop is laid on it and the operations just described are repeated, a continuation of the same forming the knitted fabric. These operations form what is known as the plain stitch, which has been previously referred to and described. Other stitches can be made with these needles by varying the method of forming them, or by other and different attachments to the machine.

(*To be continued.*)