A LABORATORY REPRODUCTION OF THE VISCOSE PROCESS

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A laboratory process for the preparation of rayon is described. The process follows the commercial method faithfully in the major details. Only commonplace chemicals and apparatus are required.

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The viscose process for the production of rayon, in addition to its being the most important commercially, is, perhaps, the most interesting from the standpoint of the reactions involved. These reactions are, in the main, the formation of the mono-sodium ester of cellulose, the subsequent formation of the sodium xanthogenate, or sodium xanthate, and finally the conversion of the xanthate back to cellulose.

The laboratory process described here is built around these three reactions, and follows the commercial process faithfully in its major details. The chemicals used are commonplace, and the apparatus is easily assembled. Furthermore, since there is no point to an attempt to equal the commercial product in the quality of the viscose silk, rigid control of humidity and temperature is unnecessary. If normal room temperature, or slightly below, is maintained throughout the experiment, the results should be satisfactory.

Experimental

Immerse a highly porous filter paper (cellulose) of about 7 cm. diameter in a 20% aqueous solution of sodium hydroxide. The mercerizing which takes place in the solution will require about three hours, and will result in the formation of the sodium complex, $[2(C_6H_{10}O_5)_x]\cdot x\text{NaOH}$. After sufficient time has elapsed, remove the mercerized paper from the caustic and press it as dry as possible with a spatula. Tear the paper into small crumbs, and place them immediately into a wide-mouth bottle (50 cc. capacity). Allow the crumbs to age for thirty-six hours in the tightly stoppered bottle.

Proper conditions of humidity are very important in the aging, or ripening process. If the mercerized cellulose is dried between filter papers or by exposure to air before it is allowed to age, the resulting product will form either an inferior grade of viscose or none at all.

After the crumbs have ripened, transfer them to a test tube, add 3 to 5 cc. of carbon bisulfide, and cork the tube tightly (do not use a rubber stopper). Within two or three days the formation of the xanthate should be complete. The crumbs have now swelled up, become rubbery and gelatinous, and orange-brown in color. The reaction which has occurred may be represented as follows:

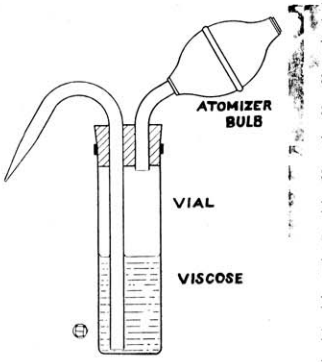
$$[2(C_6H_{10}O_5)_x]\cdot x\text{NaOH} + xCS_2 \longrightarrow [(C_6H_{10}O_5)_2\cdot C\cdot S\cdot S\cdot Na]_x$$

Remove the excess carbon bisulfide, pressing as much of the liquid out of

the crumbs as possible with a glass rod, and allow the tube to remain open for a few minutes. Then add 5 to 6 cc. of 5% sodium hydroxide solution and again stopper the tube. The formation of the viscose will require from two to three days.

After its formation is complete, the viscose, which is a colloidal emulsoid dispersion of sodium xanthate in sodium hydroxide solution, must be aged for two days. During this aging process, the viscosity of the solution decreases and reaches a minimum after about two days. The viscose is then ready to be spun.

The details of the spinning apparatus are given by the illustration.



From this wash-bottle-like affair the viscose is to be spun into a hardening bath consisting of a solution of 12% sulfuric acid and 4% sodium sulfate in water. Proceed as follows: pour the hardening bath into a shallow glass dish, such as a small developing tray. Place the nozzle of the spinner just below the surface of the hardener, and force the viscose into the bath by gently pressing the bulb. Cause the flow of viscose to be as even as possible, and move the nozzle about in the solution to prevent tangling of the thread. The crude "silk" forms as a glistening, light yellow, somewhat hollow thread. Wash it thoroughly in warm, soapy water, then rinse with pure warm water.

The crude viscose silk contains some free sulfur which can, if it is desired to continue the purification, be removed by immersing the thread for twenty minutes at 70°C. in a solution of four parts sodium sulfide in one thousand parts water. At the end of this time, remove the thread, wash it, and bleach it with chlorine or 3% hydrogen peroxide. After the bleaching is complete, wash the silk with soapy water, then pure water, and allow it to dry.