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## METHOD OF PREPARING ALKALI CELLULOSE

Francis M. Patton, Jr., Asheville, N. C., assignor to American Enka Corporation, Enka, N. C., a corporation of Delaware

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This invention relates to the manufacture of alkali-cellulose, and more particularly to a method of supporting sheets of cellulose pulp within a soaking vat during the admission of soaking lye thereto.

Preparatory to the manufacture of viscose rayon, sheets of cellulose pulp are treated with an aqueous solution of sodium hydroxide, referred to as soaking lye, in soaking vats. The soaking lye reacts with the cellulose to give sheets of alkali-cellulose, which thereafter can be shredded, aged, and carried through the remainder of the several operations that precede the formation of the viscose solution. It has been determined that the manner in which the first reaction takes place is of great importance with respect to the quality of the viscose solution and the final product. In other words, if soaking conditions are not correct, difficulties will be encountered in operations such as xanthation and filtration which will be undesirable from the viewpoint of both operating cost and product quality.

The soaking operation has been the subject of extensive investigation but the preponderance of efforts along these lines, as indicated by the patent literature available, has been directed to the chemical conditions of soaking and to the overall mechanical apparatus. In spite of the many improvements that have been developed in this art, however, there still has remained the problem of obtaining uniform soaking of the cellulose sheets.

The cellulose sheets are loaded into the soaking vats in so-called "books," or assemblages of from 10 to 30 sheets, which are inserted vertically between a series of flat perforated spacing plates adapted to maintain the books in vertical position. Inasmuch as the sheets possess low structural rigidity, they tend to bend slightly and lie against the supporting plates.

As the soaking lye is admitted to the vat, the liquid level rises gradually, submerging the several books of cellulose sheets. In the normal way of carrying out this operation, the inside and outside sheets comprising the separate books are exposed to uneven treatment.

The action of soaking lye upon cellulose sheets, in addition to causing the chemical reaction, causes a high degree of transverse swelling and a slight shrinkage in the two long dimensions of the sheets. At the bottom of the sheets, this initial swelling forces adjacent sheets against each other to such an extent that the free penetration of lye between individual sheets at the inside of the book is substantially prevented. As

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the lye continues to rise, the outside sheets of the book are thus exposed to soaking lye of substantially correct concentration, while the inside sheets contact only whatever soaking lye may slowly seep in between the tightly pressed swollen sheets.

A further complication that prevents the free passage of lye to all sheets is the so-called "slumping" of the sheets during the initial stages of filling the vat. When the lower portions of the sheets are wetted with the soaking lye, their strength is appreciably reduced. The weight of the unsupported remaining dry portions of the sheets then causes a buckling and jamming of the weakened bottom portions, so that the penetration of lye between the sheets, especially the inside sheets, is impeded still further.

It is known that cellulose, when contacted with an excess of an aqueous solution of sodium hydroxide, has a preferential absorption for the sodium hydroxide, with the result that the remaining solution has a lower concentration than the original solution. Thus, the soaking lye that passes along the sheets of pulp is lower in alkali content than the fresh soaking lye. Furthermore, the moisture content normally retained by cellulose sheets, which is about 10% based on the weight of the cellulose, further dilutes the already impoverished soaking lye.

The final result is that the outside sheets of pulp in each book are soaked in the lye bath at substantially the specified concentration of sodium hydroxide, while the inner sheets are soaked in lye that has a lower concentration with respect to the sodium hydroxide. This difference leads to non-uniformity of alkali-cellulose crumbs, less satisfactory xanthation, and considerably poorer filtration characteristics.

It is therefore an object of this invention to provide an improved method for soaking sheets of cellulose with an aqueous solution of sodium hydroxide whereby all sheets are evenly contacted with a solution of substantially uniform concentration.

It has been found possible to accomplish this objective by supporting the books of cellulose sheets at their uppermost edges during the period of initially filling the soaking vat with soaking lye, and then removing the supporting means at the completion of the filling operation.

In practicing the process, several books of sheets are deposited in a soaking vat consisting of a plurality of compartments, one book for each compartment. A sheet supporting means of any simple design is mounted above the soaking

vat and is adapted to clamp each of the books together prior to the addition of the soaking lye. As soon as the books are individually clamped together, the soaking lye is added gradually until the books of sheets are submerged after which the clamping means are released and the soaking is completed in the normal manner.

It can be seen from the foregoing description that this invention can be practiced with the use of a simple and inexpensive mechanical addition to a customary soaking vat. The results obtained by the use of this invention show a pronounced improvement in the final viscose, as illustrated by the following example:

*Example*

In a standard soaking vat, two compartments were used for a comparison of the process of the invention with the normal process. In each compartment a book of 22 sheets of cotton linters pulp sheets were place. Clamps were fastened at the upper corners of one of the two books, and in turn the clamps were affixed to the soaking vat. Before the lye was introduced, the bottom of the two books both rested on the bottom of the vat. Soaking lye was then allowed to fill the vat. During this filling the unsupported sheets exhibited the usual slumping, with lye penetration into the inside sheets being incomplete at the end of the filling period. The supported sheets, as they absorbed the lye, shrank slightly, with the result that the bottom edges of the sheets rose above the bottom of the vat, thereby enabling the passage of lye underneath the book. The sheets at the outside and the inside of the book supported from above soaked uniformly, with the lye level being the same for the outside and inside sheets.

The sheets were pressed in the normal manner, and then removed from the large press and separated. From each book were taken 5 outside and 5 inside sheets, which were then shredded separately into white crumbs in a small Baker-Perkins laboratory shredder. Samples were taken for crumb analysis, after which the remaining crumbs were processed in the normal manner into a viscose containing 7.3% cellulose and 6.8% NaOH. Samples of the viscose were taken immediately after dissolving for microscopical determination of gels and fibers. The remainder of the dissolving viscose was passed through a small filter cloth of known area at constant pressure, with the weight of filtrate being continuously recorded. A clogging constant was then obtained from the observed filtration data according to the well known law of Hermans.

The experiment was conducted in duplicate, with the results given below:

| | Sample | | | | Control | | | |
|---|---|---|---|---|---|---|---|---|
| | I | | II | | I | | II | |
| | Outside | Inside | Outside | Inside | Outside | Inside | Outside | Inside |
| White Crumb Analysis: | | | | | | | | |
| NaOH | 15.20 | 15.16 | 15.20 | 15.36 | 15.24 | 14.80 | 15.24 | 14.28 |
| Cell | 33.94 | 33.96 | 32.88 | 32.26 | 32.46 | 33.80 | 33.06 | 33.92 |
| Viscose Analysis: | | | | | | | | |
| Gels | 77 | 75 | 50 | 49 | 174 | TMC* | 174 | TMC* |
| Fibers | 44 | 28 | 23 | 24 | 38 | | 73 | |
| Clogging Factor ($\times 10^{-4}$) | 76.7 | 64.7 | 64.5 | 79.2 | 29.8 | 884.0 | 56.6 | 1,055.0 |

*Too many gels and fibers to make an accurate count.

As can be seen from the data in the above table, the differences in properties existing between the outside and inside sheets of pulp that were soaked without being supported were essentially eliminated when the pulp sheets were supported from above. This can be noticed especially in the values of the clogging factor, which were greatly higher than normal for the inside sheets that were soaked without being supported. In comparison, the clogging factor of a normally prepared viscose varies between 0.0070 and 0.0090.

It can also be seen from the above data that there is a general improvement in gel and fiber count even for the outside sheets when these sheets are supported during filling. This can be attributed to the better alignment of the sheets that are supported during filling. Without any support, the sheets often tend to float upwards or sideways during filling, and then when the sheets are subsequently pressed, there are portions of wet pulp that do not receive the intended pressure, giving rise to the so-called "wet edges" which hinder xanthation and give poor viscose quality as determined by the gel and fiber count.

It can be realized that this process of supporting the pulp sheets from the top during the filling of the soaking vat is extremely beneficial from the standpoint of improved quality. Also, since the resulting viscose has a lower clogging factor, filtration cloths do not have to be renewed as frequently, representing a saving in labor and material.

What is claimed is:

1. A method of treating a plurality of sheets of cellulose with an aqueous lye solution that comprises initially supporting a plurality of adjacent sheets of cellulose in a vertical position from their respective bottom edges in a soaking vat, exposing the sheets to an aqueous lye bath of progressively increasing height by gradually introducing lye solution into the soaking vat with resultant decrease in the vertical dimension of the sheets and concurrent with the decrease, transferring the support of the sheets from their bottom edges to the respective upper edges thereof.

2. A method of preparing alkali cellulose sheets from sheets of cellulose pulp that comprises positioning a plurality of such sheets in a soaking vat, clamping the sheets together near the top thereof to form a unit while initially supporting the sheets from their bottom edges, submerging the sheets in an aqueous lye solution by gradually filling said soaking vat with lye solution and thereafter suspending the sheets from their upper edges until they are submerged, whereafter the sheets are unclamped for the completion of the soaking operation.

3. A method of preparing alkali cellulose from sheets of cellulose pulp comprising the steps of positioning a plurality of adjacent sheets in a soaking vat in substantially vertical position, clamping the sheets together near the top thereof by means of a fixed clamp attached to said soaking vat while said sheets are being supported on the bottom of said vat, gradually supplying an

aqueous solution of sodium hydroxide to the bottom of said vat, whereby said sheets are caused to shrink slightly and become suspended from said clamp and uniform penetration of said sodium hydroxide solution is effected among the sheets, continuing to supply sodium hydroxide to the bottom of said vat until said sheets are completely submerged, and thereafter releasing said sheets from said clamp.

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