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XANTHATION PROCESS

Andrew Robertson, Richmond, Va., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

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This invention relates to a process for preparing cellulose xanthate suitable for use in the viscose process for producing rayon, cellophane, and the like, and is more particularly concerned with a process for preparing cellulose xanthate from sheets of cellulose pulp without shredding the sheets.

In the viscose process for the production of filaments, films, and the like, cellulose pulp sheets are commonly steeped in aqueous caustic solutions of mercerizing strength, pressed to remove excess solution, and shredded to form alkali cellulose crumbs. The alkali cellulose crumbs are aged in cans for several days to mature and are then xanthated with carbon bisulfide in a baratte. This conventional xanthating equipment is well-known to those skilled in the art. A good illustration of a baratte will be found on page 116 of "Synthetic Fiber Developments in Germany" by Leroy H. Smith (Textile Research Institute, Inc., New York, 1946). The pressed alkali cellulose sheets have a density of approximately 40 pounds per cubic foot when stacked neatly into a container, or a bulk density of as low as 20 pounds per cubic foot if dumped at random into a container. After shredding the sheets into crumbs, the alkali cellulose takes up at least twice as much space as the unshredded, randomly packed sheets, the bulk density becoming only about 10 pounds per cubic foot. Furthermore, when the ripened alkali cellulose crumbs are xanthated they shrink markedly and occupy only a fraction of the volume required at the start of the xanthation. This loss in the effective capacity of the baratte could be avoided or considerably reduced if high density unshredded alkali cellulose could be used in the process.

It has long been realized that it would be more economical to avoid the use of low density alkali cellulose crumbs, but no satisfactory alternative has been devised previously. It had been proposed to shred and xanthate alkali cellulose simultaneously, but this has not been commerically successful. It had also been proposed to steep a thin waterleaf paper, composed of extremely short fibers of not over 0.2 millimeter in length, and to xanthate the resulting alkali cellulose without shredding. Even if it were possible to handle such low fiber length stock, such material would not give viscose products having acceptable properties.

An object of the present invention is to provide a process for preparing cellulose xanthate from sheets of cellulose pulp without shredding the sheets. Another object of the invention is to

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prepare alkali cellulose in a high density form suitable for xanthating, thereby increasing the effective capacity of the equipment used over that achieved with low density alkali cellulose crumbs. A further object of the invention is to provide a process for xanthating high density alkali cellulose in sheet form. Other objects of the invention will become apparent from the following description and claims.

The objects of this invention are accomplished by a process which comprises steeping commercially available viscose grade wood pulp or cotton linter cellulose sheets in caustic solution to form alkali cellulose, pressing the alkali cellulose sheets to a dry press weight ratio not greater than 2.8, aging the alkali cellulose in sheet form, charging the aged alkali cellulose into a conventional rotatable baratte in sheet form, drawing a relatively high vacuum of more than 20 inches of mercury on the baratte, and then xanthating the alkali cellulose by adding carbon bisulfide and rotating the baratte to tumble the reacting alkali cellulose. Shredding or grinding blades are not used during xanthation or elsewhere in the process.

Commercially available wood pulp cellulose used for preparing cellulose xanthate is a high alpha-cellulose sulfite pulp having an average fiber length of 1 to 2 millimeters and regularly furnished in sheets having an air-dry basis weight of 0.15 to 0.20 pound per square foot. Cotton linters, which may also be used as the cellulose raw material, are essentially alpha-cellulose having an average fiber length of 0.5 to 2.5 millimeters and regularly furnished in sheets having a basis weight of 0.08 to 0.15 pound per square foot.

The alkali cellulose sheets may be quartered or cut or torn into even smaller pieces, when such pieces are accommodated more readily in the aging cans or baratte, but the pieces are still in the high density sheet form and not in the low density crumb form. This effectively increases the capacity of existing equipment, eliminates the shredding equipment formerly required, and also saves the power previously consumed in the shredding operation.

In view of the unsuccessful attempts of the prior art, it is surprising that alkali cellulose can be xanthated in the dense sheet form. Nevertheless, when the critical conditions set forth are followed, the xanthated product will be crumbly, yellow-orange in color, and equal in quality to the best cellulose xanthate produced by the conventional process with alkali cellulose crumbs. Evaluation of the quality included tests of solu-

tion preparation, filter-ability, spinning performance, and properties of the regenerated cellulose products.

On the other hand, if an attempt is made to use wood pulp or linter alkali cellulose at press weight ratios of 3.0 or more, a gummy, balled-up mass of non-uniformly xanthated cellulose will be found in the baratte when it is opened. Not only is the result unsatisfactory, but a considerable amount of time will be spent in cleaning up the mess. By press weight ratio is meant the final weight of the stepped and pressed sheet divided by the original weight of air dry cellulose sheet charged into the steeping press.

The xanthation will be started after the alkali cellulose has been subjected, in the baratte, to an absolute pressure of 10 inches of mercury or less, produced by drawing a relatively high vacuum of more than 20 inches of mercury on the baratte. Preferably, but not necessarily, xanthation will be conducted with an excess of carbon bisulfide, the xanthation reaction being ended prior to exhaustion of the carbon bisulfide by removing the excess from the zone of reaction. The amount of excess carbon bisulfide may be from 3% to 150% more than is required for the desired reaction end-point, other conditions being the same as for conventional xanthation except that the time cycle will be reduced.

By means of this invention a substantial saving is made by elimination of the shredding step. The power saving alone is considerable, but in a new plant the invention will also eliminate a substantial investment in machinery and in building capacity because the alkali cellulose will be aged and xanthated in a dense form. This volume space requirement can be reduced substantially 50% and baratte charges can be increased at least 50%. All of this economy is achieved without sacrifice of quality in the final product. Numerous tests have shown that filterability of the viscose prepared in accordance with this invention is at a commercially acceptable level and, when wood pulp is used as the source of cellulose, it is at least as good as that previously obtained by the conventional process, and the time required for mixing, blending, etc. is not noticeably altered if other conditions remain the same.

As different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited to the specific processes disclosed except as defined in the appended claims.

What is claimed is:

1. A process for preparing cellulose xanthate which comprises steeping high alpha cellulose sheets having an average fiber length of 0.5 to 2.5 millimeters and a basis weight of 0.08 to 0.20 pound per square foot in caustic solution to form alkali cellulose, pressing the alkali cellulose to a relatively dry press weight ratio not greater than 2.8, aging the alkali cellulose in sheet form, subjecting the unshredded alkali cellulose to a vacuum of more than 20 inches of mercury and then adding carbon bisulfide to the relatively dry unshredded alkali cellulose and tumbling the mixture to xanthate the cellulose.

2. A process for preparing cellulose xanthate which comprises steeping sheets of high alpha-cellulose pulp having an average fiber length of 1 to 2 millimeters and a basis weight of 0.15 to 0.20 pound per square foot in caustic solution to form alkali cellulose, pressing the alkali cellulose to a press weight ratio not greater than 2.8, aging the alkali cellulose in sheet form, and then vacuum xanthating the relatively dry unshredded alkali cellulose and tumbling the alkali cellulose during xanthation using a vacuum of more than 20 inches of mercury.

3. A process for preparing cellulose xanthate which comprises steeping sheets of cotton linters having an average fiber length of 0.5 to 2.5 millimeters and a basis weight of 0.08 to 0.15 pound per square foot in caustic solution to form alkali cellulose, pressing the alkali cellulose to a press weight ratio not greater than 2.8, aging the alkali cellulose in sheet form, and then vacuum xanthating the relatively dry unshredded alkali cellulose using a vacuum of more than 20 inches of mercury and tumbling the alkali cellulose during xanthation.

ANDREW ROBERTSON.

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