Sept. 8, 1964 R. L. MITCHELL ETAL 3,148,106

PULP REFINING

Filed Jan. 29, 1964 2 Sheets-Sheet 1

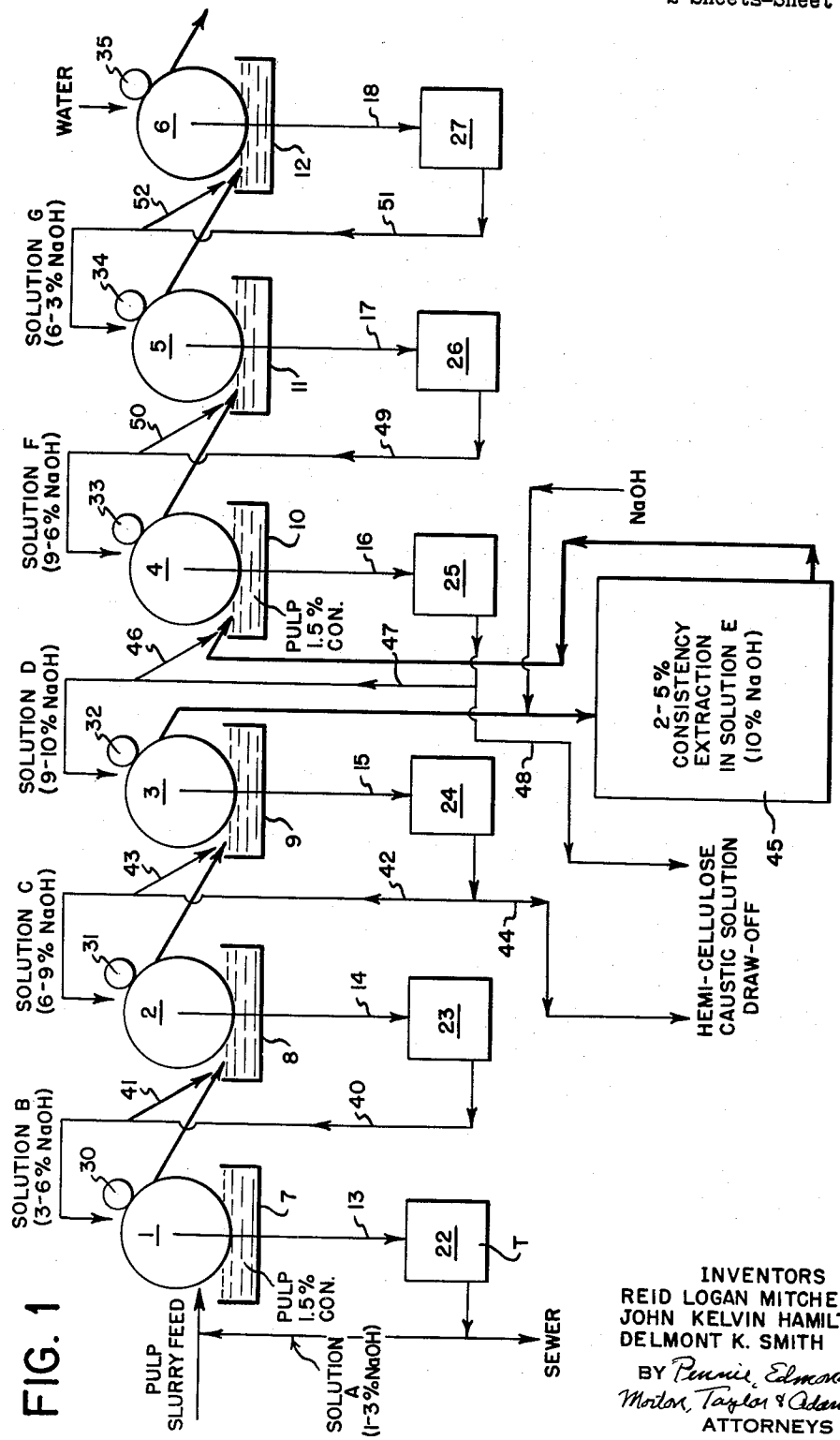


FIG. 1

INVENTORS
REID LOGAN MITCHELL
JOHN KELVIN HAMILTON
DELMONT K. SMITH

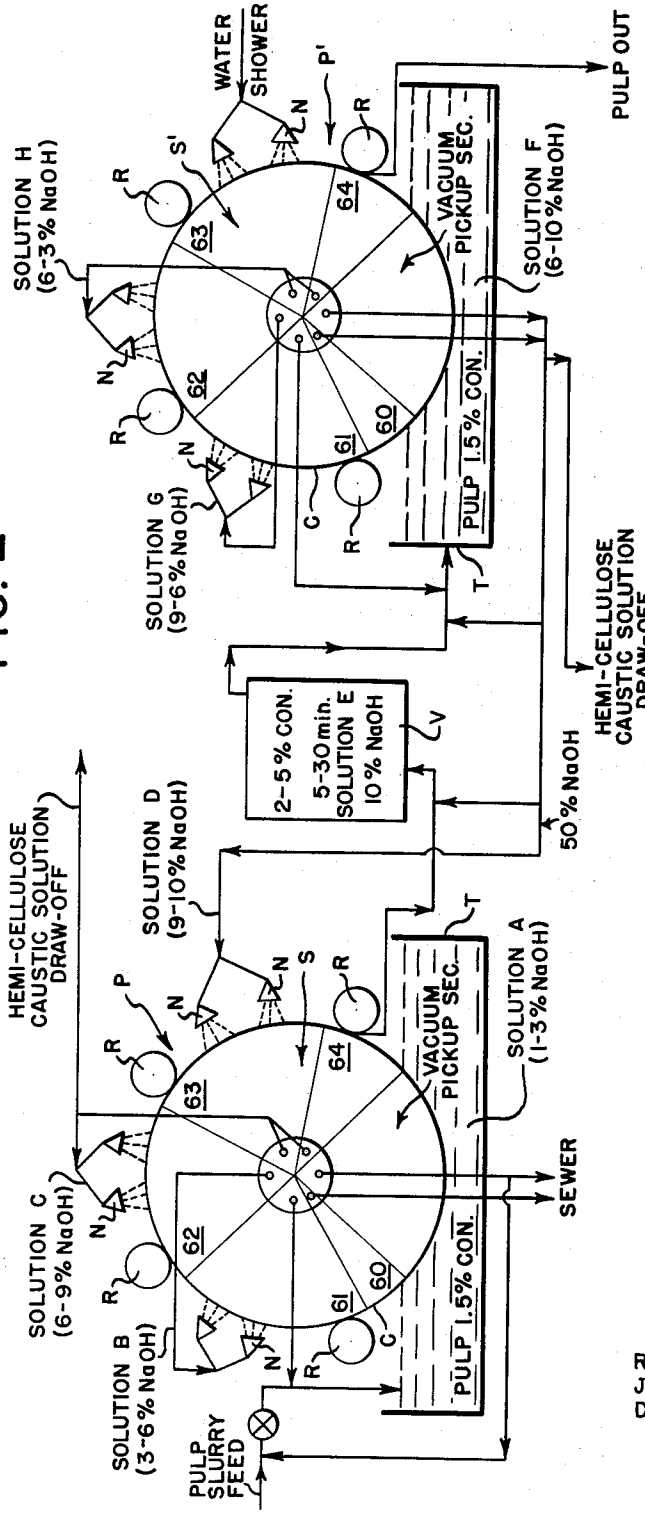
BY Pennie, Edmonds, Morton, Taylor & Adams
ATTORNEYS

Sept. 8, 1964 R. L. MITCHELL ETAL 3,148,106

PULP REFINING

Filed Jan. 29, 1964 2 Sheets-Sheet 2

FIG. 2



INVENTORS
REID LOGAN MITCHELL
JOHN KELVIN HAMILTON
DELMONT K. SMITH

BY Pennie Edmonds, Morton, Taylor & Adams
ATTORNEYS

United States Patent Office 3,148,106 Patented Sept. 8, 1964

3,148,106

PULP REFINING

Reid Logan Mitchell, Morristown, N.J., John Kelvin Hamilton, Shelton, Wash., and Delmont K. Smith, Longmeadow, Mass., assignors to Rayonier Incorporated, Shelton, Wash., a corporation of Delaware

Filed Jan. 29, 1964, Ser. No. 340,908
10 Claims. (Cl. 162—18)

This invention relates to the cold-caustic refining of wood pulp to produce high-alpha cellulose, and has for its object the provision of an improved process consisting of a series of extractions, first with cold-caustic solutions of successively increasing concentrations, then with a special low-consistency slurry stage extraction at maximum caustic concentration, and then with a series of cold-caustic solutions of successively decreasing concentrations ending with a suitable water wash. The concentration of the caustic solutions in the several stages is varied from about 1 to 3% at the start to about 9 to 10% at the stage of highest concentration and then progressively decreases in stages of caustic removal, followed by a water wash. It is a characteristic of the process of the invention that the caustic solution on the pulp at a particular stage is displaced by the succeeding caustic solution, both in progressively increasing the concentration and progressively decreasing the concentration. It is a further charactertistic of the process that a part of the caustic solution rich in dissolved hemicellulose is continuously removed from the system at a place where the solution has a high ratio of hemicellulose to caustic. The temperature is preferably maintained between 5° and 40° C. with the preferred range being about 15° to 30° C.

The prior publications and patents pertaining to cold-caustic refining, and variations of cold and hot caustic refining, are voluminous, and several variations of cold-caustic refining have been and now are being used to produce high-alpha cellulose.

In the usual cold-caustic refining process the wood pulp is treated one or more times with a concentrated caustic solution at relatively low temperature, say room temperature or below. The objective of the refinement is to obtain by selective solution fractionation on the wood pulp the removal of short-chain cellulose and noncellulosic carbohydrates as well as other impurities with a minimum of destructive action on the long-chain cellulose. The result theoretically should be a comparatively high yield of a uniform and highly purified product, but this process as practiced up to now has its drawbacks. In conventional processes, large volumes of strong caustic solution are required from which it is difficult to recover the spent chemical. The high swelling of the cellulose in the presence of cold concentrated caustic solution and its resultant "slippery" nature and tenacity for the caustic solution make it difficult to handle with conventional washing equipment. Caustic is preferentially sorbed by cellulose, drainage of the swollen cellulosic material is very slow and displacement of the caustic is nonuniform, resulting in incomplete washing usually coupled with some clumping of fibers as well as reprecipitation of dissolved hemicellulose on the refined product. As a result of these difficulties, and particularly with respect to the nonuniformity of the treatment of the pulp and high requirement of chemical, full utilization of the cold caustic process to give extremely high alpha pulps on a commercial scale has not been achieved.

The invention employs a number of caustic solution extractions with interstage increases in pulp densities and caustic displacements which not only give a superior and very highly purified product but a surprising economy in the use of caustic. Moreover, the process very effectively removes the undesirable materials of the pulp

without appreciably degrading the cellulose, resulting in a high-alpha cellulose in high yield that is free of stuck fiber clumps or knots.

The initial step in the improved process of the invention consists in slurrying the unextracted wood pulp with a relatively dilute caustic solution, say, from 1 to 3% at a low consistency of from 1.5 to 5%. This slurry is concentrated to a consistency of about 10 to 35% by suitable mechanical means such as a vacuum washer-press roll combination or a centrifuge. A stronger solution of caustic, preferably about 3 to 6%, is then flushed through the high consistency pulp on the washer in such a manner as to displace the residual weaker solution. The pulp is concentrated to a consistency of 10–35% and is flushed with a 6 to 9% caustic solution to displace the residual 3 to 6% caustic solution. The pulp is again concentrated to a higher consistency of about 10 to 35% and is then flushed with about 9 to 10% caustic solution to displace the residual solution of from 6 to 9% caustic. At this stage a fraction of this displaced caustic solution which is rich in the extracted impurities, mainly hemi-cellulose, is bled out of the system. The pulp is again concentrated to a consistency of about 10 to 35%. At this point the special low-consistency extraction stage is used in the process and the high density pulp with it residual strong caustic is slurried with additional caustic solution of about 10% concentration to give a consistency of from about 2 to 5%. This slurry in concentrated caustic solution is agitated for from 5 to 30 minutes, after which the caustic is gradually removed by successive displacements with caustic solutions of decreasing concentrations, finally ending with a water wash. In this caustic removal the concentrated caustic is first removed by pressing to a high consistency in a mechanical manner and the residual caustic is displaced by a solution of about 9 to 6% caustic, followed by another mechanical pressing and displacement with a weaker solution of about 6 to 3% caustic, further interstage pressing and finally displacement with a water wash. The foregoing operations are carried out at a temperature between 5° to 40° C., preferably in the range of from 15° to 30° C.

In the course of the foregoing process, the weak caustic solution as first applied selectively dissolves most of the xylose and galactose-type hemicellulose as well as some of the glucomannan hemicelluloses from the wood pulp. These are removed for the most part with the removal of the weak caustic solution as the slurry is concentrated in the high density interstage step and the final traces are displaced by the stronger caustic used to flush out the residual weaker caustic in the ensuing stage. No intervening water wash is used, no xylose or galactose-type hemicelluloses and soluble glucomannan hemicelluloses are reprecipitated on the cellulose and this, coupled with the dilute midpoint slurry treatment, leads to uniformity of extraction and completeness of removal. In their turn the stronger caustic stages (6 to 10 percent) swell the microfibrils of cellulose and dissolve most of the so-called "resistant" short chain glucomannans and degraded "beta" cellulose fractions. Removal of the xylose and galactose-type hemicelluloses and soluble glucomannan hemicelluloses improves drainage and porosity of the pulp blanket. The concentrated caustic stages and the low consistency interstage treatment at maximum caustic strength essentially completes the removal of the more difficultly soluble "resistant" glucomannans, as well as most of the remaining resistant xylose-type and galactose-type hemicelluloses, which are soluble only in concentrated caustic solution. After the low consistency stage with the highest concentration of caustic, the gradual and uniform removal of the caustic solution from the high level of concentration by stages through displacement with

weaker caustic solutions is also very important. Gradual reduction of caustic with interstage pressing to high consistency greatly reduces the chance of reprecipitation of residual dissolved hemicellulose and importantly prevents the swollen fibers from adhering to each other to form clumps or, as they are sometimes called, "stuck," "compacted" fibers, or "fish eyes." The latter have long plagued the industry.

It is extremely difficult to achieve the highest levels of cellulose purity (i.e., 98–99 alpha level) with any reasonably low usage of caustic because many of the materials needing removal, although soluble in 10% NaOH at say 25° C., do not remain soluble as caustic is washed out of the pulp blanket. It is one thing to solubilize these materials and quite a more difficult task to flush them away and remove them from the system without excessive use of caustic solution, and/or reprecipitation of hemicellulose (causing clumping of fibers).

The system of the invention accomplishes this objective. A key point in this system which helps conserve caustic is the unusual mid-point withdrawal of waste materials plus the feedback of extraction solutions to early stages thereby selectively sorbing (and conserving) caustic on the cellulose building up to maximum concentration while at the same time solubilizing easily soluble hemicelluloses. This sorption of caustic on the cellulose from feedback of extracts permits withdrawal of an effluent toward the center of the system which is not only relatively lean in caustic and relatively rich in hemicellulose, but is relatively low in volume. The intermediate dilute slurry stage is also a key feature because it permits a polishing type of extraction in which the last traces of soluble material are solubilized and allowed to leach out from the swollen fibers. Actually a good part of solubilized impurities have been removed already from the system prior to this dilute polishing stage, and the final solubilized residues can then be subsequently gradually flushed out in stepwise displacements in such manner as to avoid undue reprecipitation and clumping or matting of the individual pulp fibers.

Removal of hemicellulose prior to the most critical extraction stage helps to free up the fibers and improves draining and uniformity of caustic removal in the interstage pressing operations on the downhill cycle of caustic removal. The interstage pressing on the uphill side when caustic is being built up is not as critical because fibers have not yet reached maximum swelling. Mercerization and "curling" of the fibers in the low consistency strong caustic stage also substantially facilitates ease and uniformity of drainage during the downhill cycle of caustic displacement and removal.

Although the present system resembles a counter-current system in some aspects, it is actually quite different. In the stepwise buildup of caustic to a peak concentration and then the reverse in the stepwise removal of caustic to prevent clumping, hemicellulose removal from the system occurs near the center and not at the end of the sequence as would be the case with true countercurrent. A true countercurrent system could not really work in this type of purification, since impurities that had been solubilized in the strongest caustic stage would be reprecipitated as caustic concentration is subsequently decreased.

The efficiency of the present system is keyed to achievement of high interstage densities or consistencies. In as much as caustic swells the cellulose fibers to a degree dependent on both concentration and temperature, it is important to balance out the system to use as high consistency as can be obtained at each interstage.

The accompanying drawings illustrate apparatus which may be used advantageously in carrying out the process of the invention in which:

FIG. 1 is a diagrammatic representation in side elevation of one arrangement of apparatus, and

FIG. 2 is a side elevation of another embodiment of apparatus.

The apparatus illustrated in FIG. 1 comprises a series of vacuum drum washers **1**, **2**, **3**, **4**, **5**, and **6** each of which is rotated and suspended over a washer vat **7**, **8**, **9**, **10**, **11** and **12**. The washers have peripheral screen (not shown) over which the pulp is treated and are operated under a vacuum, the solution passed through the pulp on the screens being removed through pipes **13**, **14**, **15**, **16**, **17** and **18** which discharge into sealed effluent tanks **22**, **23**, **24**, **25**, **26** and **27**. Each drum has a press roller **30**, **31**, **32**, **33**, **34** and **35** which compresses the pulp over the screens on the drums. The low consistency extraction vessel **45** is arranged to receive pulp from drum **3**, and concentrated caustic solution.

It will be understood that the various drum washers of the apparatus illustrated are operated under an effective vacuum, that the pulp is agitated in the washer vats, and that when the pulp layer picked up by the screens of the drum washers passes under the press rollers the liquid is removed both by compression and suction to increase interstage consistency.

In order to simplify the following discussion of the process the caustic solutions will be referred to as:

Solution A, 1 to 3% NaOH; solution B, 3 to 6% NaOH; solution C, 6 to 9% NaOH; solution D, 9 to 10% NaOH; solution E, about 10% NaOH; solution F, 9 to 6% NaOH; and solution G, 6 to 3% NaOH.

In carrying out a process of the invention in the apparatus of FIG. 1 a slurry of unextracted wood pulp such as a normal sulfite or a kraft pulp of 90–96 alpha (the higher the alpha the harder it is to achieve further purification) is fed into vat **7** and is slurried at a low consistency of about 1.5% in solution A of 1 to 3% NaOH. (Hereinafter consistency refers to the percent weight of oven dry pulp in the slurry.) A layer of the pulp is picked up on the screen of drum **1** and as the drum turns clockwise some of this weak solution is sucked out of the pulp. Solution B which is passed from tank **23** through pipe **40** is showered over the pulp layer to displace residual solution A, and as the screen passes under roller **30** the pulp is compressed and much of solution B is removed to increase the consistency to 10 to 35%. The solution sucked through the pulp on the screen of drum **1** consisting of solution A and some solution B is passed through pipe **13** to tank **22** from which it may be returned in part to the pulp feed or discarded to the sewer. The pulp from washer **1** is passed into vat **8** and also some solution B is passed through pipe **41** into vat **8**.

The pulp is picked up on the screen of drum **2** and most of the solution B is sucked through the pulp. Solution C, received through pipe **42** from tank **24**, is showered onto the pulp to displace the residual solution B and as the drum passes under roller **31** the pulp is compressed to a consistency of 10 to 35%. Solution B and some of solution C which are sucked through the pulp on drum **2** are passed through pipe **14** to tank **23**. The pulp from drum **2** with the residual solution C on the pulp is passed into vat **9** and some solution C is also passed through pipe **43** into vat **9**. The pulp is picked up on the screen of drum **3** and much of the solution is sucked through the pulp. Most of solution C is removed by this suction but the residual solution is displaced by solution D, received through pipe **47** from tank **25**, which is showered on the pulp. As the pulp on the screen of drum **3** passes under roller **32** the pulp is compressed to a consistency of from 10 to 35% and some of solution D passes along with solution C through pipe **15** into tank **24**.

The solution removed from the pulp on drum **3** has a high hemicellulose to caustic ratio. Some of this solution is continuously removed from the system to prevent an excessively high accumulation of hemicellulose. A convenient place to remove a part of this solution is to bleed a fraction of the solution from tank **24** through

pipe 44. Additional removal of solutions with a high hemicellulose to caustic ratio may also be taken from tank 25 through pipe 48. Usually from 80 to 95 percent of the hemicellulose caustic solution should be removed from the system at these two points.

The pulp on drum washers 4, 5 and 6 are treated successively with weaker caustic solutions and is given a final wash with water as now to be described.

The pulp from drum 3 together with the residual solution D is passed into tank 45 to which some 50% NaOH solution "buckup" is added to obtain the desired 10% caustic solution concentration and the pulp is slurried at a consistency of from 2 to 5%. The pulp from tank 45 is passed into vat 10 to which some of solution D is added through pipe 46. The pulp at a consistency of 1.5% in vat 10 is picked up on the screen of drum 4 and most of the solution is sucked out. The weaker solution F, received through pipe 49 from tank 26, is showered onto the pulp to displace the contained solution and as the pulp passes under roller 33 the consistency is increased to from 10 to 35%. The expressed solution F and the solution sucked from the pulp are passed through pipe 16 to tank 25. The pulp from drum 4 together with the residual solution F go to vat 11, and some additional solution F is passed through pipe 50 into vat 11.

The pulp is picked up on the screen of drum 5 and most of the contained solution is sucked out. Solution G, passed through pipe 51 from tank 27, is showered onto the pulp on the screen of drum 5. This displaces most of the residual solution F and as the pulp passes under roller 34 the pulp is pressed to a consistency of from 10 to 35%. The removed solution F with some solution G are passed through pipe 17 to tank 26. The pulp from drum 5 containing only residual weak caustic solution G is passed into vat 12, and some solution G is passed through pipe 52 into vat 12. The pulp is picked up on the screen of drum 6 and most of the contained caustic solution is sucked out of the pulp and passed through pipe 18 into tank 27 from which it is passed through pipe 51 to drum 5. Fresh water is showered on the pulp to displace the residual caustic solution and as the pulp passes under roller 35 most of the water is squeezed out and the purified pulp is removed from the system for further processing. The drum 6 may be segmented to remove separately the caustic solution G and the wash water to prevent dilution of solution G.

The process of the invention may also be practiced with the apparatus illustrated in FIG. 2 which comprises two segmented vacuum washer units P and P' each of which has a screened cylinder C rotatable over a fixed segmented structure S comprising a vacuum pickup section and vacuum sections 60, 61, 62, 63 and 64. This arrangement of apparatus comprises a solubilizing stage, an extraction stage and a flushing and washing stage.

At spaced positions on the periphery of the cylinder rollers R are arranged to apply pressure on the pulp picked up on the screened cylinder C. Each press roll unit is partially immersed in a tank T in which pulp is slurried.

In order to simplify the following discussion with reference to the process carried out in the apparatus of FIG. 2 the caustic solutions are referred to as:

Solution A, 1 to 3% NaOH; solution B, 3 to 6% NaOH; solution C, 6 to 9% NaOH; solution D, 9 to 10% NaOH; solution E, 10% NaOH; solution, F, 8 to 10% NaOH; solution G, 9 to 6% NaOH, and solution H, 6 to 3% NaOH.

In carrying out a process of the invention in the apparatus of FIG. 2, a slurry of unextracted wood pulp such as sulfite pulp is fed into the tank T of unit P and is slurried at a low consistency (con.) of about 1.5% in solution A and a layer of this pulp is picked up on the screen over the vacuum pickup section. (Hereinafter consistency refers to the percent weight of oven dry pulp in the slurry.) As the screen C turns clockwise some of this weak caustic soda solution is sucked out of the pulp in the vacuum pickup section and in section 60 and this weak solution is either discharged to a sewer or returned to the initial slurrying operation. When the screen passes under the roller R over section 61 the pulp is compressed and much of the solution A is removed to increase the consistency to from 10 to 35%. As the screen and its layer of pulp move under the spray nozzles N a caustic solution is applied to the pulp to displace the residual caustic solution A. All of the caustic solution which enters section 61 is preferably returned to the slurry feed. This stage of the operation is controlled to displace the weak caustic solution A with stronger caustic solution B while avoiding the undue entry of the stronger caustic solution B into section 61.

As the pulp layer moves to section 62 and passes under the roller N thereover a sufficient portion of the caustic solution B is extracted to again increase the consistency to from 10 to 35%. When this extracted pulp passes under the nozzles N it is showered with a caustic solution C to displace the residual caustic solution B. The displaced caustic solution B is showered on the pulp in section 61.

As the pulp layer passes under roller R of section 63 the caustic solution C is squeezed out to a consistency of about 10 to 35%. Thereafter more concentrated solution D, 9 to 10% caustic, is applied by the nozzles N of section 63 to flush out the caustic solution C applied at section 62. This last caustic solution D is pressed out when the pulp passes under roller R of section 64 and the pulp mat is sent to a slurry vessel V where the pulp is slurried in a 10% caustic solution at a consistency of 2 to 5% for from 5 to 30 minutes. The solution removed from the pulp in either section 63 or section 69 has a high hemicellulose to caustic ratio and is wholly or partially removed from the system to separate from 80 to 95% of the hemicellulose from the system.

The 10% caustic solution of vessel V is effluent from washer P' with some 50% caustic added. This slurry is then passed into the slurry tank T of unit P'. Additional solution of about 10% caustic is added, some of which may come from section 61, to reduce the slurry to a consistency of about 1.5% and from this slurry the pulp is picked up on the vacuum section and as the screen moves clockwise over vacuum section 60 this strong caustic solution F is sucked out and this solution F from the vacuum pickup section is returned to earlier stages of the process when required and, if necessary, the caustic is replenished by addition of a 50% sodium hydroxide solution.

As the pulp layer passes under the roller R of unit P' of section 61 much of the caustic solution F is squeezed out to a pulp consistency from 10 to 35% and then the pulp passes under the nozzles N where the pulp is showered with solution G which may advantageously be received from section 62 to displace the residual caustic solution F. Some of the solution F initially sucked out of the pulp on the drum and some of the solution from section 60 which contain hemicellulose may be removed from the system as shown on the drawing. When the pulp passes under the roller R of section 62 the caustic solution G is squeezed out to a consistency of 10 to 35% and then the pulp passes under the nozzles N of this section where the pulp is showered with caustic solution H in an amount sufficient to displace the residual caustic solution G. The layer of pulp then passes under the roller R of section 63 to press out much of the caustic solution H to a consistency of from 10 to 35%. As the pulp layer passes under the nozzles N of section 63 the pulp is showered with water to wash out the residual weak caustic solution H. The pulp then passes under roller R of section 64 where the water is removed as much as possible and the pulp is ready for further processing. It will be understood that in the apparatus of FIG. 2 the various sections are operated under an effective vacuum

and that when the pulp layer passes under the rollers the liquid is removed both by compression and suction.

It will be apparent that the various lines and direction arrows indicating the passage of solution from one stage to another are merely illustrative of what may be done to utilize most effciently the sodium hydroxide contained in the various solutions. In actual operations these solutions are passed through valve controlled pipes and are passed into intermediate solution containers where the various concentrations are adjusted and where the depleted sodium hydroxide may be made up by introducing solutions of, say, 50% sodium hydroxide.

It is to be understood that the process may also be carried out with a multi-stage continuous, centrifugal extractor having perforated concentric extractor portions. Such apparatus is available and may be provided with means for applying the caustic solution, concentrating the pulp, displacing residual caustic, and removing caustic solution, washing means, and the connection and adaptation of an interstage slurry flushing means.

The following is an illustrative example of a process in accordance with the invention:

The partially bleached stock entering the process has an alpha cellulose content of approximately 96% and a consistency near 15%. The water present is displaced by a solution containing approximately 3% caustic and 1% hemicellulose and the pulp mat is pressed to 33% consistency. Caustic at this concentration and a temperature of 25° C. (this temperature is used throughout) has so little solvent power for hemicellulosic material that it is neither able to dissolve such materials from the entering stock nor to prevent the precipitation of a portion of the hemicelluloses onto the fibers. At this stage, there is then transfer of caustic and also a net gain in the hemicellulosic material associated wtih the stock. Displacement of the 3% caustic with a 6% caustic solution containing 1 to 1.5% hemicellulose followed by pressing to 33% consistency results in a slight swelling of the fibers and the dissolution of enough hemicellulosic material to increase the purity of the stock above its original level. Further swelling of the cellulose and the dissolution of additional hemicellulose and some short chain cellulosic material is obtained when the 6% caustic is displaced with 9% caustic and the mat is pressed to 33% consistency. The solution at this stage contains a high hemicellulose to caustic ratio and some of it is continuously removed from the system. The pulp, containing at this point approximately 98.5% alpha cellulose, is slurried with 10% caustic at 3% consistency and agitated for a period of 20 minutes. This stage increases the purity of the pulp to approximately 99.3% alpha cellulose and insures uniform extraction of the fibers. After thickening and pressing to 33% consistency, the 10% caustic is displaced with 7% caustic and pressed to 33% consistency. Some solution containing hemicellulose and caustic may be removed from the system for discard after the slurrying with 10% caustic. A 3% caustic solution essentially free of hemicellulose is used to displace the 7% caustic; followed by pressing to 33% consistency. The pulp is then washed with water to remove the remaining caustic. The resultant product is of uniform purity, approximately 99.3% alpha cellulose, and is free from stuck or compacted fibers. Samples of pulp of the invention and pulp not so treated were made into viscose, and spun into tire cords. These cords were given standard fatigue flex tests. The average fatigue life for a control sample (not given a cold caustic extraction) was 350 minutes, whereas pulp given the foregoing treatment had a fatigue life of about 380 minutes.

The degree of refinement attained in the above illustration of the process is merely an example of the capabilities of the process. The amount of refinement (and the yield loss) can be controlled by altering the maximum caustic strength, the temperature, and the hemicellulose level carried in the system.

In the foregoing illustration it should be noted that preferential displacement of hemicellulosic material and preferential retention of caustic are obtained by the use of high interstage densities. In this manner there is obtained a high hemicellulose content in the 6 and 9% caustic solutions separated from the stock before the 10% caustic extraction. The maximum amount of hemicellulose is obtained with the minimum of caustic by withdrawing at these points the amount of hemicellulose effluents necessary to maintain a given hemicellulose level. This is a highly desirable feature from the standpoint of economy in that less washer facilities would be required in a commercial unit.

Several undesirable effects occur if there is not a gradual decrease in the caustic concentration of the solutions following the low consistency 10% caustic extraction. If the caustic strength is lowered for example from 10% to 1% there are many compacted or stuck fibers formed which give rise to nonuniform sheet and poor processability. Secondly, if the hemicellulose content of the more dilute displacing caustic solutions is not kept at a low level, especially for caustic concentrations of about 7 and below, there is a precipitation of dissolved hemicellulose on the swollen pulp. Thus, for example, the alpha cellulose content of 99.3% which is obtained from the low consistency 10% NaOH extraction is lowered to the range of 98.5% by the too rapid dilution of the caustic solution strength from 10 to 1% in the presence of 1% hemicellulose. In addition to the lowering of the degree of refinement and uniformity of purification, large amounts of stuck fibers are also obtained.

Advantages of the invention include:

(1) Uniform treatment of all the cellulosic fibers in the wood pulp at each stage of the process.

(2) Gives extremely high levels of purity (i.e., 98–99+alpha) without presence of stuck fibers. High density replacement of liquor alternated with low consistency treatments without the use of water washes, etc., leading to complete removal of solubilized material from the product, particularly when coupled with the special agitated low consistency extraction interstage step where retention time is 5 to 30 minutes at maximum caustic concentration.

(3) Gives very conservative caustic usage. The high densities developed between each stage plus the feedback of extracting solutions facilitate maximum drainage and uniform flushing of treating solutions and insure use of minimum volumes of caustic solution as well as help prevent fish eyes, stuck fibers, etc.

(4) The process can be made essentially continuous and automated. By appropriate collection and distribution of expressed solution, efficient flow of pulp and caustic solutions is obtained using relatively simple equipment and with easy segregation of minimum amounts of drainage solutions high in hemicellulose and low in caustic.

This application is a continuation-in-part of our application Serial No. 118,452, filed June 20, 1961, and now abandoned.

We claim:

1. In the cold-caustic refining of wood pulp at a temperature of from 5° to 40° C. to produce high-alpha cellulose wherein the wood pulp is slurried with aqueous caustic solutions in stages to solubilize and remove hemicelluloses, short-chain celluloses and other impurities, the improvement which comprises slurrying the pulp with a very dilute cold caustic solution at a low consistency, concentrating the pulp to a consistency of 10 to 35%, displacing the residual caustic solution of the pulp with a 3 to 6% caustic solution, concentrating the pulp to a consistency of from 10 to 35%, displacing the residual 3 to 6% caustic solution with a solution containing up to about 10% of caustic, removing most of this solution containing up to 10% of caustic from the pulp to give a consistency of from 10 to 35%, withdrawing a part of this last-mentioned solution which contains hemicelluloses

and other impurities from the process, then slurrying the pulp in a caustic solution of about 10% at a dilute consistency of from 2 to 5%, displacing the 10% caustic solution through a series of displacement extractions each of which is followed by displacement with a caustic solution of lower concentration, and then washing the pulp with pure water, thereby removing impurities without reprecipitation of the same on the pulp fibers and without clumping or "fish eye" formation resulting in a very highly purified high-alpha cellulose.

2. In the process of claim 1 carrying out the first slurry at a consistency of from 1.5 to 5% with a caustic solution of from 1 to 3%.

3. In the cold-caustic refining of wood pulp at a temperature of from 5° to 40° C. the improvement which comprises slurrying pulp with about a 3% caustic solution at a consistency of from 1.5 t o 5%, concentrating the pulp by mechanical means to extract a major portion of the caustic solution, displacing the residual caustic solution with a solution of from 3 to 6% caustic, increasing the consistency by mechanical thickening to from 10 to 35%, displacing the 3 to 6% caustic solution with a solution containing up to about 10% caustic, then increasing the consistency by mechanical extraction to from 10 to 35% to remove most of the solution containing up to about 10% caustic, removing a part of this last-mentioned solution which contains hemicelluloses and other impurities from the process, adding additional caustic solutions of about 10% concentration, subjecting the pulp to agitation for from 5 to 30 minutes at a low consistency with caustic solution of about 10% caustic, then effecting a removal of the caustic solutions in a series of extractions and displacements with caustic solutions of successively decreasing caustic concentrations, and washing the pulp with water.

4. In the process of claim 3 increasing the caustic concentration in steps in each of which the pulp is compressed to remove a major part of the caustic solution, and then a more concentrated caustic solution is fed back and showered on the compressed pulp to displace the weaker residual solution thereon.

5. In the process of claim 3 decreasing the caustic concentration in steps in each of which the pulp is compressed to remove the major part of the caustic solution, and a less concentrated caustic solution is added to the compressed pulp to displace the more concentrated caustic solution.

6. In the process of claim 3 carrying out the process at a temperature of from 10 to 30° C.

7. In the cold-caustic refining of wood pulp at a temperature of from 5° to 40° C. to produce high-alpha cellulose the improvement which comprises slurrying a cellulose pulp with about a 1 to 3% caustic solution at a consistency of from 1.5 to 5%, compressing the slurry to remove the solution to a consistency of from 10 to 35%, increasing the caustic concentration on the pulp to about 10% in a succession of solution additions and extractions in which a weaker caustic solution is displaced with a fed back stronger caustic solution and the consistency is increased to from 10 to 35%, removing a part of the solution containing up to about 10% of caustic which contains hemicellulose and other impurities from the process, adding 10% caustic solution to reslurry, subjecting the pulp to agitation for from 5 to 30 minutes at a lower consistency with a caustic solution of about 10%, then effecting a removal of the caustic solution from the pulp by successively displacing the stronger caustic solution with a weaker caustic solution, and washing the pulp with water.

8. In the process of claim 7 increasing the caustic concentration successively in steps in each of which the pulp is compressed to remove a major part of the caustic solution followed by displacement of the residual caustic solution with stronger fedback caustic solution.

9. In the cold-caustic refining of wood pulp at a temperature of from 5° to 40° C. to produce high-alpha cellulose wherein the wood pulp is slurried with aqueous caustic solutions in stages to solubilize and remove hemicelluloses, short-chain celluloses and other impurities, the improvement which comprises slurrying the pulp with a very dilute cold caustic solution at a low consistency, concentrating the pulp to a consistency of 10 to 35%, displacing the residual caustic solution of the pulp with a 3 to 6% caustic solution, concentrating the pulp to a consistency of from 10 to 35%, displacing the residual 3 to 6% caustic solution with a solution containing up to about 10% of caustic, removing most of this solution containing up to 10% of caustic from the pulp to a consistency of from 10 to 35%, withdrawing a part of this last-mentioned solution which contains hemicelluloses and other impurities from the process, then slurrying the pulp in a caustic solution of about 10% at a consistency of from 2 to 5%, concentrating the pulp to a consistency of from 10 to 35%, displacing the residual caustic solution of the pulp with a 6 to 9% caustic solution, withdrawing a part of this last-mentioned solution which contains hemicelluloses and other impurities from the process, and displacing the 6 to 9% caustic solution through a series of displacement extractions each of which is followed by displacement with a caustic solution of lower concentration, and then washing the pulp with pure water, thereby removing impurities without reprecipitation of the same on the pulp fibers and without clumping or "fish eye" formation resulting in a purified free-fiber high-alpha cellulose.

10. The improved process of producing high-alpha cellulose from wood pulp which comprises the following sequence of operations at temperatures below 40° C.:

(1) slurrying the pulp at a low consistency in a weak solution of less than 3% of caustic,
(2) extracting the weak caustic solution from the pulp to a consistency of from 10 to 35%,
(3) displacing the residual weak caustic solution by addition of a stronger caustic solution of from 3 to 6% caustic,
(4) extracting sufficient of the stronger caustic solution to increase the consistency to from 10 to 35%,
(5) removing from the process a part of the extracted strong caustic solution which contains hemicellulose and other impurities from the pulp,
(6) displacing the residual stronger caustic solution with a solution of about 10% caustic,
(7) extracting a substantial proportion of the 10% caustic solution,
(8) returning additional 10% caustic solution to the process to displace said strong caustic solution removed with hemicellulose,
(9) slurrying the pulp in a solution of about 10% caustic at a low consistency,
(10) extracting a substantial proportion of the 10% caustic solution from the slurried pulp to increase the consistency to from 10 to 35%,
(11) removing the residual 10% caustic solution by pressing and displacement with 6% to 9% caustic solution, removing the 6% to 9% caustic solution by pressing and displacement with a solution of 3% to 6% caustic solution, and
(12) washing the pulp with water.

References Cited in the file of this patent

FOREIGN PATENTS

570,075 Germany ---------- June 21, 1933

OTHER REFERENCES

Jayme: Refining of Pulp from Paper Trade Journal, pp. 37–42, May 26, 1938.