

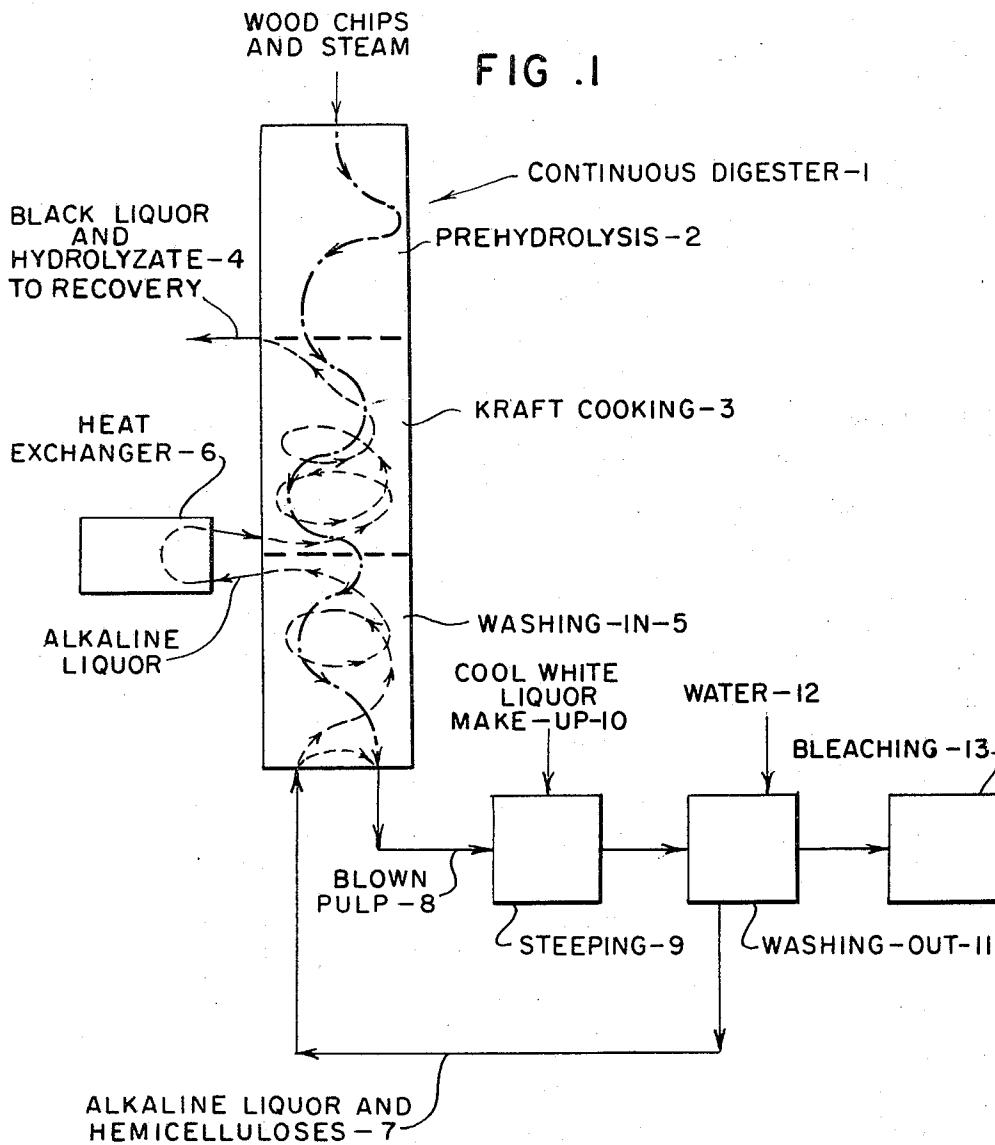
Dec. 27, 1966

A. W. BRINKLEY, JR., ETAL
CONTINUOUS DIGESTION AND PURIFICATION
WITH RECIRCULATION OF LIQUOR

3,294,623

Original Filed Feb. 23, 1962

3 Sheets-Sheet 1



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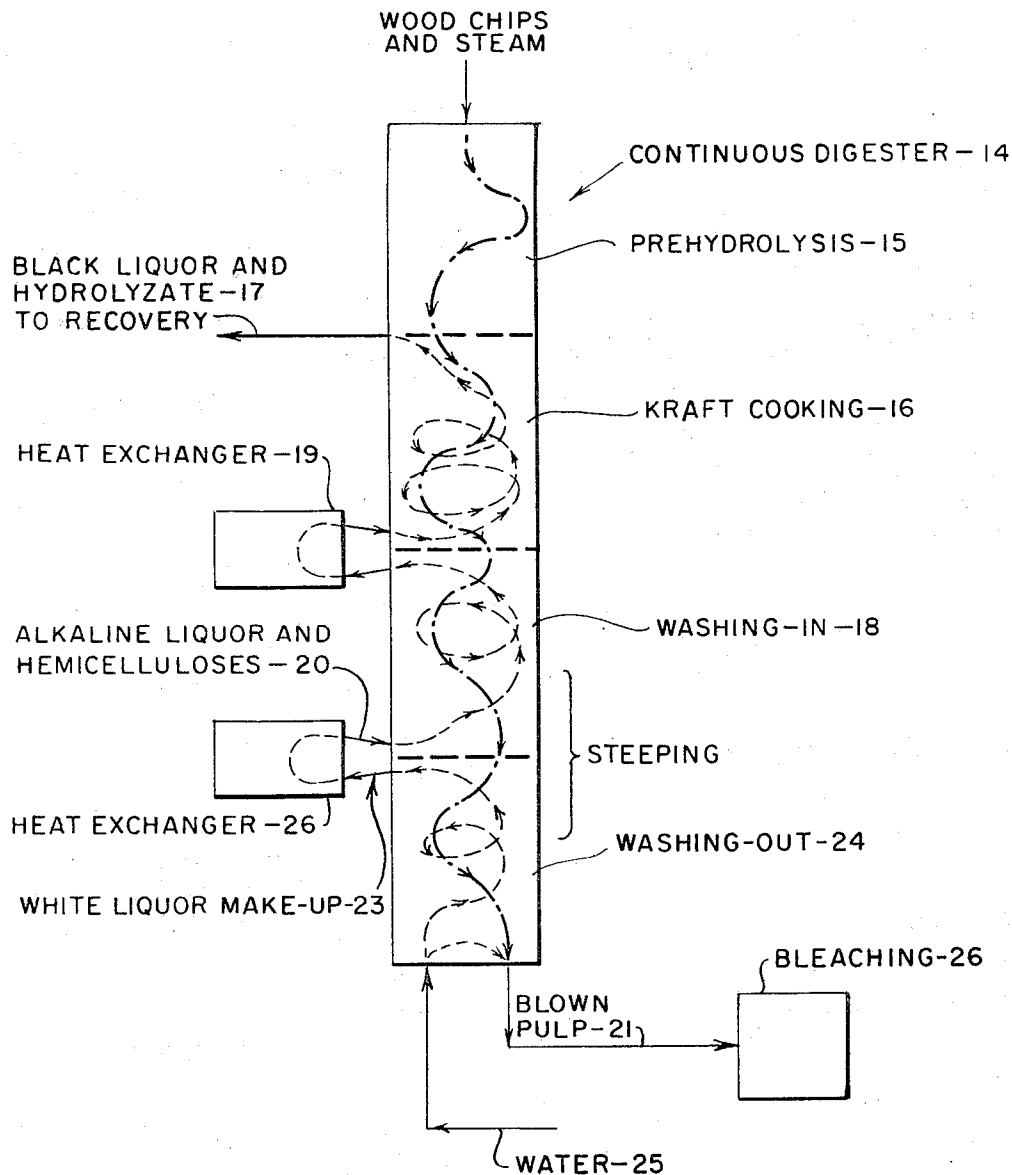
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FIG. 2



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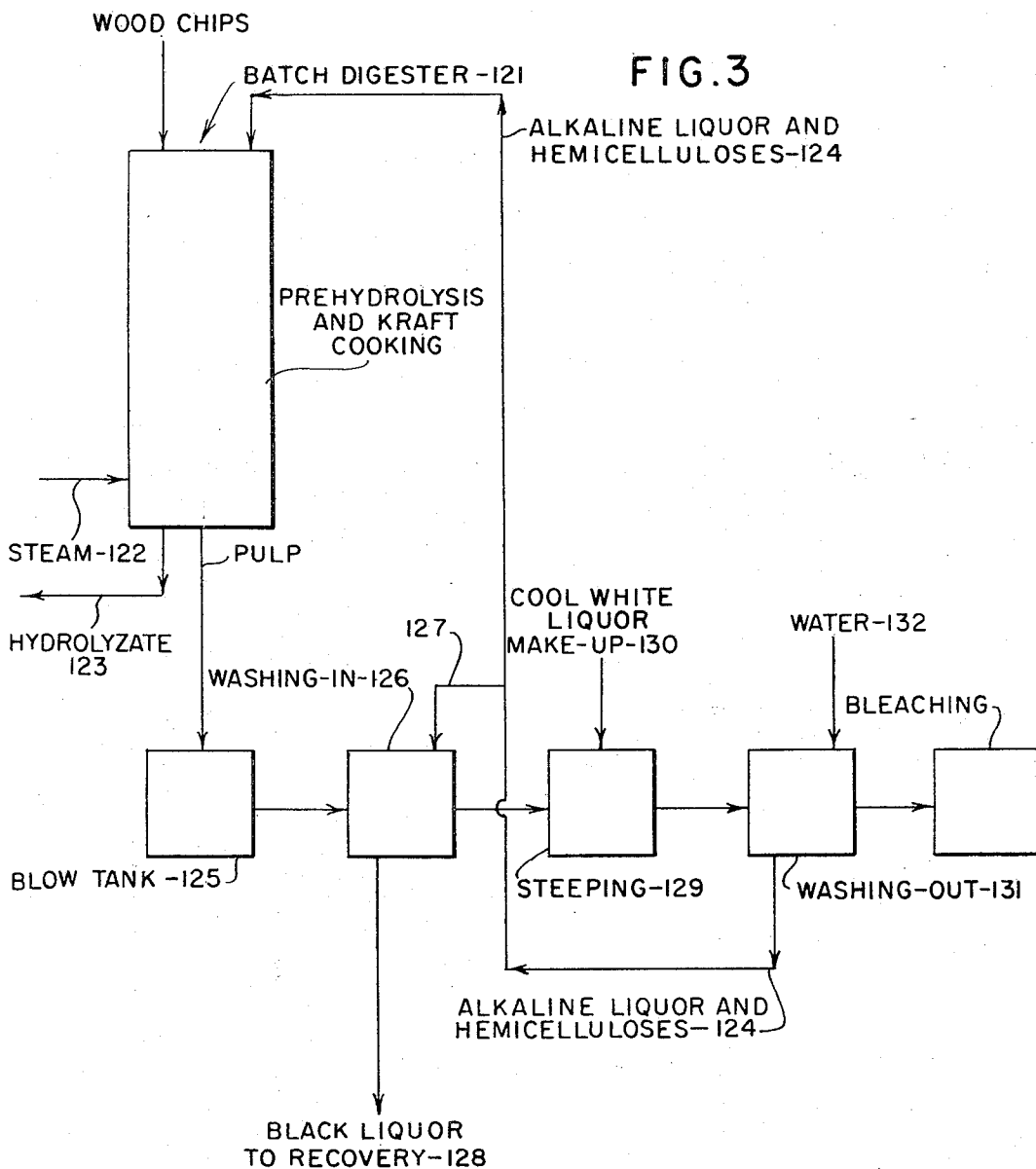
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3,294,623

**CONTINUOUS DIGESTION AND PURIFICATION
WITH RECIRCULATION OF LIQUOR**

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Continuation of application Ser. No. 176,489, Feb. 23, 1962. This application Feb. 23, 1966, Ser. No. 529,291
15 Claims. (Cl. 162—19)

This application is a continuation of copending Serial No. 176,489, filed February 23, 1962, now abandoned which was a continuation-in-part of Serial No. 810,877, filed May 4, 1959, and of co-pending Serial No. 494,196, filed October 8, 1965. This invention relates to a process for preparing cellulose pulp which is particularly useful in the preparation of dissolving grade wood pulps for conversion to produce rayon and other cellulose derivative products.

Constant demands are made upon the rayon industry for rayon yarn and cord of greater strength, durability, and other desirable characteristics, and this in turn requires manufacture of dissolving grade pulps to increase quality at competitive prices.

Since production of pulp for conversion involves a substantial loss in cellulose yield resulting from the high degree of pulp processing and refinement necessary for this use and which yield is otherwise available for other pulp uses such as papermaking, it is a principal problem of the industry to produce, at competitive price levels, conversion pulps from which the rayon manufacturer can obtain maximum yields of rayon having maximum strength, fatigue resistance, and other desirable features.

Accordingly, it is the overall objective of the present invention to provide a superior dissolving grade pulp. More specifically, the object is to provide process steps in the preparation of pulp for conversion which improve the quality and the overall characteristics which are desirable in such pulps, including maintaining and increasing rayon cellulose yield and rayon strength and fatigue characteristics, while at the same time substantially reducing production costs.

Another object is to improve the removal of hemicelluloses and other impurities in pulp processing and at the same time to avoid mercerization and undue lowering of viscosity.

Another object is to make available a process by which cheaper and more plentiful pulpwoods may be processed to produce conversion pulp of a standard currently required or better.

These and other objects will be further discussed in more detail in the description of the invention, the practice of which is supplementally illustrated in attached FIGURE 1.

FIGURES 1 and 2 are simplified flow diagrams of two different ways of performing the process of this invention showing essentials, but omitting details including many of the pumps, valves, meters, recording instruments and other equipment, the adaptation to the process of which will be apparent to those skilled in the art once the process itself is understood.

Generally speaking, the process of this invention is applicable, prior to bleaching, in any one of the alkaline processes for treating cellulosic fibrous material such as wood chips which contain organic material including lignin and hemicelluloses to produce pulp and is preferably used as an improvement in the sulfate or kraft process. It includes cooking and, preferably, also a partial hydrolysis of the cellulosic fibrous material before cooking. Such cooking can be continuous, using a Kamyr digester, as described at the February 1965 meeting of

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TAPPI (Technical Association of the Pulp and Paper Industry) in New York City. It also includes treatment, immediately following cooking, of pulp which contains a substantial amount of alkali in the form of black liquor from cooking.

The part of the process which follows cooking involves treatment of the pulp with an alkaline liquor which is later used for cooking and is conveniently designated "post treatment." Therefore, as will further appear, the total process including cooking involves very little alkali (soda) loss and the need for only a comparatively small amount of make-up liquor, so that great economy results from its use.

The process of the invention broadly comprises, in addition to cooking, three sequential steps which combine removal of the black cooking liquor from the pulp with further pulp refinement.

The first step following cooking comprises washing the cooked chips or "pulp" with the alkaline solution in such a manner that the black liquor which is in the pulp is displaced and removed from the pulp and replaced in the pulp by the alkaline solution. This step is, therefore, conveniently designated as "washing in," because the alkaline solution is washed into the pulp.

The alkaline solution can contain one of or any combination of the common alkalies or caustics such as the hydroxides, sulfides, or carbonates of sodium, lithium, or potassium. Since the preferred practice of this invention is associated with the kraft or sulfate process, the alkali applied to the pulp is kraft white liquor which has been used previously, as will be described, and which comprises sodium hydroxide, sodium sulfide, and sodium carbonate. The white liquor can be fortified or mixed with another alkaline solution such as a strong sodium hydroxide solution containing up to about 75% by weight of the hydroxide. In the preferred treatment, the kraft cooking stage is somewhat like the countercurrent cooking described in U.S. Pat. No. 3,097,987, issued July 16, 1963, utilizing a Kamyr digester, in that the wood chips flow down the digester and are contacted by progressively stronger alkaline liquor flowing up the digester. In the patented process, the alkaline liquor is added at the bottom of the kraft cooking zone, and the chips continue their flow down the digester through a wash zone where the alkali become progressively weaker until the chips reach the bottom of the digester, where wash water is added.

In the presently claimed process, instead of adding strong alkaline liquor at the bottom of the cooking zone, it is added, or "washed in," at the bottom of the digester washing zone, from which it flows upward displacing alkaline black liquor from the pulp. Then, at the top of the washing zone, the strong alkaline liquor is withdrawn from the digester, heated, and reinjected to begin cooking. The black liquor which is finally withdrawn from the top of the cooking zone contains some excess applied alkaline liquor and dissolved hemicelluloses from the pulp and is returned to the black liquor recovery system where it is concentrated and burned to produce heat and to recover chemicals for the pulp mill. The pulp emerging from the "washing in" step in the bottom of the digester contains another part of the applied alkaline solution.

The second step after cooking comprises steeping the pulp in the alkaline solution added during the "washing in" step, to which make-up alkaline liquor can be added, preferably in a steeping tank for a predetermined period of time at a selected temperature and alkali concentration, and is conveniently designated as the "steeping" step. Alternatively, this step can be carried out within the digester itself by proper adjustment of conditions (i.e., temperature and alkali concentration) in the "washing in" step.

Surface active agents of certain types can also be added to the steeping step to aid in the removal of impurities such as resins which otherwise would not be dissolved by the alkali alone.

The third step after cooking comprises washing the pulp with water in such a manner that the alkaline solution with the pulp from the steeping step is displaced and removed from the pulp as liquid effluent and replaced in the pulp by the water. This step is, therefore, conveniently designated as the "washing out" step, because the alkaline solution is washed out of the pulp, preferably by passing it over a series of pulp washers and washing it countercurrently with water or by adding the water at the bottom of the digester washing zone. The liquid effluent from the "washing out" step is recycled to the "washing in" and "steeping" steps and subsequent to the "washing in" step is used in the cooking process.

Results of the process are that a superior dissolving grade pulp is produced which will yield a higher percentage of rayon (of the order of 98.5-99%) and greater rayon yarn strength at a cost in the range of three to four dollars per ton less than the cost of similar pulp heretofore produced without benefit of the present invention, and this is an outstanding gain. The countercurrent cooking gives higher purity than was heretofore possible, since the cellulose material is contacted with successively increasing alkali concentration. (It is well known that conventional cooking, in which the alkali concentration progressively decreases, tends to redeposit hemicelluloses.) In addition to improved purity, greater economy results from adding all of the alkaline liquor at or near the bottom of the digester and displacing it into the chips.

Other results are that the process herein described and claimed consistently lowers the permanganate number of the pulp which means that the pulp will bleach more easily, so that bleaching chemical requirements, principally for chlorine, are reduced. This, together with the increased overall yield that is obtained from the wood, more than offsets the cost of the make-up alkali (soda) required for the post treatment. Then too, the post treatment process avoids mercerization and undue lowering of viscosity.

Since the liquid effluents of the present process are re-used in the overall pulp process, the only chemical raw material cost involved is that of the alkali necessary for make-up to replace what is carried out of the process with the pulp and which is small in proportionate amount—about 30 pounds of soda per ton of finished machine-dried pulp.

In short, the process of this invention results essentially in a substantial improvement in rayon strength, fatigue, and other quality characteristics of the pulp, in its chemical analyses including rayon cellulose content and all at a substantial reduction in process cost.

The pulp to be treated according to the process of this invention is preferably first subjected to a partial prehydrolysis and then cooked in the same or a separate digester in accordance with treatment sometimes known in the art as the prehydrolyzed kraft process. See, for example, co-pending Serial No. 309,839, filed September 18, 1963. Briefly stated, the prehydrolysis is treatment of the prepared wood chips in a digester tank with steam or hot water or both at elevated temperature and pressure, to which, in some cases, an acid such as sulfuric may be added, and which is in any event a partial acid hydrolysis which solubilizes some of the hemicelluloses, including pentosans, and other impurities in the chips.

In cooking (digesting) by the kraft process in accordance with the present invention, the kraft or sulfate white liquor, comprising sodium sulfide, sodium hydroxide, and sodium carbonate, is then introduced to the chips in the digester and cooking proceeds for a period of time at selected elevated temperatures (above 212° F.) and pressures in the usual manner, except that a countercurrent cooking process is used, to dissolve lignin and other materials and to yield pulp. The liquid effluent from the cook-

ing process is black liquor or spent cooking liquor which contains substantial quantities of dissolved wood substances and alkali.

The accompanying drawings serve to illustrate the process. FIGURES 1 and 2 illustrate ways of carrying out the process of the present invention, whereas FIGURE 3 illustrates, for comparative purposes, a related process claimed in co-pending application Serial No. 176,489, filed February 23, 1962.

Referring to FIGURE 1, wood chips or other cellulosic material to be treated is continuously charged into a digester 1 which may be a single vessel or may consist of more than one vessel. A preferred digester is constructed by Kamyr and has provisions for prehydrolysis, kraft cooking, and washing continuously in one vessel. The chips are prehydrolyzed at elevated temperatures and pressure in the top, or "prehydrolysis" zone 2 of the digester. At the bottom of the "prehydrolysis" zone 2, the chips contact the upward flowing black liquor from the "kraft cooking" zone 3. The spent black liquor and the hydrolyzate formed during prehydrolysis are ultimately extracted together from the digester through strainers 4, and the combined stream is processed by conventional methods to recover heat and chemicals for reuse in the process. The chips flowing down the "kraft cooking" zone 3 encounter increasingly stronger alkaline liquor flowing up the digester. This alkaline liquor is heated to the desired cooking temperature by extracting the alkaline liquor at the bottom of the "cooking" zone, which is the top of the "washing in" zone, heating this alkaline liquor in a heat exchanger 6, and re-injecting this heated alkaline liquor into the digester. The zones referred to are zones within the hydraulic system contained by the digester vessel and such zones are distinguished, one from another, by conditions of temperature and alkali concentration.

As the chips continue their flow down the "washing in" zone, black liquor formed during cooking is washed out of them and displaced up the digester by strong alkaline liquor which is added at the bottom of the digester 7. Since strong alkaline liquor is washed into the chips, this bottom zone of the digester is called the "washing in" zone. Part of the strong alkaline liquor flows up the digester, displacing black liquor from the chips, and after heating, cooking the chips to form pulp and more black liquor. Another part of the strong alkaline liquor is extracted, or "blown," with the chips from the bottom of the digester 8. In this extraction, or "blowing," the chips are disintegrated into individual fibers, or "pulp."

The pulp is next transferred to a "steeping" tank 9 where the pulp is steeped with the alkaline white liquor it acquired in the "washing in" step. Additional alkaline liquor, or white liquor, make-up may be added 10 to the pulp as it enters the steeping tank.

In the "washing out" step, the alkaline white liquor is washed out of the pulp on the washers 11, which may be conventional vacuum drum washers, by displacing it with water 12, and the hemicelluloses and other impurities are recirculated with the used white liquor to the "washing in" zone at the bottom of the digester. These hemicelluloses and other impurities eventually find their way out of the system with the black liquor 4 and are burned for recovery of heat. The washed pulp is ready for bleaching 13.

The process, as illustrated in FIGURE 2, does not differ substantially from that shown in FIGURE 1, except that steeping and "washing out" are carried out in the bottom portions of the digester instead of in a separate steeping tank and washers. As the chips flow down the "washing in" zone, black liquor formed in the cooking zone is washed out of them and displacing up the digester by strong alkaline liquor which is added at the bottom of the "washing in" zone 18. (Since strong alkaline liquor is washed into the chips, this intermediate zone of the digester is called the "washing in" zone.)

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These zones are adjoining, or contiguous, and each of them is distinguished by conditions of temperature and alkali concentration. The strong alkaline liquor flows up the digester, displacing black liquor from the chips, and after heating, cooking the chips to form pulp and more black liquor.

In the lower part of the "washing in" zone 22, the chips are steeped with the alkaline white liquor they acquired in the "washing in" step. Additional alkaline liquor, or white liquor, 23 is added to the alkaline liquor and hemicelluloses which are washed out of the pulp at a lower level in the "washing out" step. This fortified liquor 20 is cooled in a heat exchanger 26 and added back to the digester where it flows up through the "washing in" zone.

In the "washing out" step, the alkaline white liquor is washed out of the pulp in the washing zone at the bottom of the digester 24 by displacing it with water 25, and the hemicellulose and other impurities are recirculated with the used white liquor through the heat exchanger for cooling, then to the "washing in" zone at the next highest level in the digester. These zones are adjoining, or contiguous, and each of them is distinguished by conditions of temperature and alkali concentration. These hemicelluloses and other impurities eventually find their way out of the system with the black liquor 17 and are burned for recovery of heat. In accordance with common practice, one or more conventional vacuum drum washers can also be utilized following the "washing out" zone of the digester for complete washing of the pulp, and the liquor washed out of the pulp on the conventional washers can be used as wash water 25 at the bottom of the digester. The washed pulp is ready for bleaching 26.

For comparison, FIGURE 3 shows a way of conducting the process claimed in co-pending application Serial No. 176,489, filed February 23, 1962. Such process can be carried out in either a batch digester or continuous digester. Wood chips or other cellulosic material to be treated are charged into a digester 121. In the batch system, steam is admitted 122, and the chips are prehydrolyzed at elevated temperature and pressure. At the completion of prehydrolysis, the hydrolyzate formed is withdrawn 123.

In the kraft cooking step which follows (in FIGURE 3), an alkaline white liquor, and in some cases an additional weaker alkaline black liquor, is added 124 to the prehydrolyzed chips. Steam is added 122, and the chips are cooked at elevated temperatures and pressure. At the completion of the cooking step, the chips are discharged or "blown" from the digester into a blow tank 125. In "blowing" the chips are disintegrated into individual fibers or "pulp."

In the "washing in" step, the weak alkaline black liquor contained in the pulp is washed out on the washers 126, which may be conventional vacuum drum washers, by displacing it with strong alkaline white liquor 127, which is essentially the same as the strong alkaline white liquor 124 used to cook the chips. The weak alkaline black liquor 128 which is displaced is processed by conventional methods to recover heat and chemicals for reuse in the process.

The pulp is next transferred to a "steeping" tank 129, where the pulp is steeped with the alkaline white liquor it acquired in the "washing in" step. Additional alkaline liquor, or white liquor, make-up may be added 130 to the pulp as it enters the steeping tank.

In the "washing out" step of the process shown in FIGURE 3, the alkaline white liquor is washed out of the pulp on the washers 131, which may be conventional vacuum drum washers, by displacing it with water 132, and the semicelluloses and other impurities are recirculated with the used white liquor, partly to the digester and partly back through the "washing in" step.

It may readily be seen that the process of FIGURES 1 and 2 represents a considerable advance over that of FIGURE 3, eliminating at least one set of displacement

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washers, giving improved utilization of the alkaline liquor, and, because the chips are contacted by increasingly stronger alkaline liquor in cooking, resulting in improved cellulose purity. When the process is carried out in accordance with FIGURE 2, the steeping tank and several additional displacement washers can be eliminated.

Example I

In a typical operation according to the process of the present invention and utilizing the means portrayed in attached FIGURE 1, gum pulpwood chips are prehydrolyzed in a continuous digester for a period of sixty minutes at a pressure of 105 pounds per square inch, corresponding to a temperature of 341° F.

At the bottom of the prehydrolysis zone, the chips contact the weak alkaline black liquor, which is flowing up the digester countercurrently to the chips. This black liquor and the hydrolyzate formed during prehydrolysis are extracted through strainers 4, and heat and chemicals are recovered from the combined liquor by conventional methods.

About two hours are required for the chips to pass through the kraft cooking zone, which is maintained at about 331° F. As the chips flow down the digester, non-cellulosic materials are reacted with and displaced by the increasingly strong alkaline cooking liquor, which reaches a maximum concentration at the bottom of the cooking zone of about 60 grams of active soda (Na_2O) per liter. Temperature is controlled by extracting the relatively cool alkaline liquor from the top of the "washing in" zone, heating the liquor, and reinjecting it into the digester.

Strong alkaline white liquor with a soda concentration as apparent NaOH of about 110 grams per liter and with a temperature of about 120° F. is added to the bottom of the digester. Part of this liquor flows up the digester, displacing the weaker black liquor. Its temperature gradually increases and finally at the top of the "washing in" zone, the liquor is extracted and heated to the full cooking temperature of about 331° F.

The other part of this strong alkaline white liquor is blown with the pulp from the digester. Make-up white liquor is introduced at 10 in an amount of 1157 pounds of active soda as Na_2O per ton of air-dried finished pulp, at 0.94 pound of active soda as Na_2O per gallon of white liquor in a volume of 1230 gallons per ton of air-dried pulp.

The pulp is passed into the steeping tank 9 where its temperature is adjusted and timing is arranged so that the pulp is held in the steeping tank 9 at a temperature of 120° F. for one hour. Volumes and concentrations in the tank 9 are adjusted so that the pulp consistency is about 3% and the soda concentration as apparent NaOH is 120 grams per liter.

The pulp from the steeping tank 9 is passed over the washers 11 with wash water introduced 12.

Example II

In a typical operation according to the present invention and utilizing the means portrayed in FIGURE 2 attached, gum pulpwood chips are prehydrolyzed in a continuous digester for a period of sixty minutes at a pressure of 105 pounds per square inch, corresponding to a temperature of 341° F.

At the bottom of the prehydrolysis zone, the chips contact the weak alkaline black liquor, which is flowing up the digester countercurrently to the chips. This black liquor and the hydrolyzate formed during prehydrolysis are extracted through strainers 17, and heat and chemicals are recovered from the combined liquor by conventional methods.

About two hours are required for the chips to pass through the kraft cooking zone, which is maintained at about 331° F. As the chips flow down the digester, non-cellulosic materials are reacted with and displaced by

the increasingly strong alkaline cooking liquor, which reaches a maximum concentration at the bottom of the cooking zone of about 60 grams of active soda (Na_2O) per liter. Temperature is controlled by extracting the relatively cool alkaline liquor from the top of the "washing in" zone, heating the liquor, and reinjecting it into the digester.

At the bottom of the "washing in" zone, the chips contact wash liquor which is flowing up the digester counter-currently to the chips. This wash liquor contains alkali which has been washed from the chips in the "washing out" zone. Temperature is controlled to about 120° F. by extracting the wash liquor from the top of the "washing out" zone, cooling the liquor, and reinjecting it into the digester 20.

Make-up white liquor is introduced at 23 in an amount of 1157 pounds of active soda as Na_2O per tone of air-dried finished pulp, at 0.94 pound of active soda as Na_2O per gallon of white liquor in a volume of 1230 gallons per ton of air-dried pulp. When this fortified and cooled liquor is reinjected into the digester, the soda concentration in the chips reaches about 120 grams per liter as apparent NaOH and the temperature is about 120° F. This liquor flows up the digester, displacing the weaker black liquor. Its temperature gradually increases and finally, at the top of the "washing in" zone, the liquor is extracted, heated to the full cooking temperature of about 331° F., and returned to the same level within the digester.

Instead of using a separate steeping tank as in Example I and FIGURE 1, sufficient retention time is provided in the digester at the lower part of the "washing in" zone for steeping to occur at essentially a temperature of 120° F. and a soda concentration of 120 grams per liter apparent NaOH.

Water is added to the bottom of the digester 25. Part of this water flows up the digester displacing the strong alkaline liquor and hemicelluloses. The other part of this water is blown with the pulp from the digester 21.

More generally, while increasing the prehydrolysis time decreases the amount of resistant pentosans (hemicelluloses) present in the pulp, the overall yield of the pulp treated by the present process decreases as the prehydrolysis time is increased, probably because of hydrolytic degradation of the cellulose. Since rayon strength may also be reduced by over-prehydrolysis, the hydrolysis time should be adjusted for optimum desired conditions as to reduction of pentosan content on the one hand, and hydrolytic degradation and reduced yield on the other. The adjustment of prehydrolysis time will, of course, depend on the grade and type of pulpwood being processed.

A volume of wash water and a volume of displacement have been used at each wash stage in the post treatment of Example I and FIGURE 1 which was equivalent to 1.1 to 1.5 times the volume of liquid remaining in the pulp at 12% consistency. This may be adjusted in mill operation and should be that which minimizes soda losses and make-up requirements. If the displacement volume is too high, the amount of soda sent to the evaporators and the amount of fresh white liquor required for make-up will be higher than necessary, because an excess of the amount of alkaline white liquor necessary to displace the black liquor in the "washing in" step will end up as liquid effluent to recovery 4. If the displacement volume is too low, the soda lost with the pulp might become excessive, because of failure to displace it completely and recover it in the "washing out" step.

While it has been found that the preferred conditions for treatment of the pulp in the steeping tank 9 are a concentration of 120 grams per liter of apparent sodium hydroxide at 120° F. for a period of one hour, effective steeping time may vary in range from 5 to 120 minutes, but is not critical. Practical steeping temperature may vary in range from 70° to 180° F., but is not critical. Higher steeping temperature requires a higher liquor con-

centration. A practical operating concentration of steeping liquor may vary from 50 to 150 grams per liter as apparent NaOH, but is not critical. The practical operating consistency of the pulp during steeping may be between 1% and 6% and is preferably 3%, but is not critical. When the steeping is carried out in accordance with Example II and FIGURE 2, the operating consistency is on the order of 16%.

A strong aqueous caustic solution (25% sodium hydroxide by weight) may be used to fortify the mill white liquor used in the post treatment process, and as a rapid method of controlling the alkali concentration during the steeping step in the event of an upset. The strong caustic solution used may have a concentration between 10 and 75% sodium hydroxide by weight.

The concentration and temperature used in steeping depends upon such factors as maximum concentration available in the fresh white liquor, the minimum concentration that could be tolerated in the reclaimed liquor for cooking, the minimum temperature that could be maintained during steeping and the efficiency of displacement on the washers. In general, the desired rayon cellulose content of 98.5% (bleached) is obtained whenever the concentration in grams per liter apparent sodium hydroxide and the temperature in degrees F. are numerically the same, for example, 120 grams per liter and 120° F.

Pulps treated with the processes of this invention generally bleach to a higher brightness than do the regular pulps. This process is similar to that claimed in application Serial No. 176,489, filed February 23, 1962, in this respect. Other results are that our process consistently lowers the permanganate number of the pulp which means that the pulp will bleach more easily, so that bleaching chemical requirements, principally for chlorine, are reduced. This together with the increased overall yield that is obtained from the wood more than offsets the cost of the make-up alkali (soda) required for the treatment.

Indeed, it has been discovered that, because of the reduced bleaching chemical requirements, post treated pulp can be bleached economically to high brightness and cleanliness and with a definite improvement in rayon strength and fatigue properties by omitting the chlorination stage and subsequent caustic extraction stage and by using a minimum of hypochlorite, relying instead on one or more stages of chlorine dioxide to purify the pulp. Such a bleaching system can improve the rayon quality of non-post-treated dissolving pulps, but, because of the higher bleaching chemical requirement of these pulps compared to post treated pulps, the costs may be excessively high and the pulp could not be bleached to the same levels of brightness and cleanliness.

In a typical example of the improvements realized by this process, post treated pulp is given an acid treatment at 3.1 pH and 3.0% consistency for 40 minutes. The pulp is next washed on a drum-like washer and neutralized with caustic.

The pulp is then bleached with about 0.4% chlorine dioxide based on the pulp at 10.0% consistency and 150° F. for 190 minutes, after which the pulp is washed.

The pulp is then extracted with about 0.2% caustic soda, based on the pulp, at 10.0% consistency and 180° F. for 70 minutes, after which the pulp is washed.

The pulp is then bleached with about 0.1% chlorine dioxide based on the pulp, at 10.0% consistency and 160° F. for 190 minutes, after which the pulp is washed.

The pulp is then treated with hypochlorite stages as necessary to control the final viscosity, after which the pulp is treated with sulfur dioxide to 2.9 pH at 120° F. and 6.5% consistency for 45 minutes. The bleached pulp is then washed and is ready for further processing.

What is claimed is:

1. A process of cellulosic pulp manufacture, purification, and preparation for bleaching in which cellulosic fibrous material is continuously cooked with an alkaline liquor to produce cellulosic pulp and spent cooking liquor

suitable for recovery and in which all of the alkaline liquor is brought together with the cellulosic fibrous material after having first been displaced by water from previously produced cellulosic pulp ready to be bleached, having second been used to steep previously produced cellulosic pulp prior to its being made ready for bleaching by resort to such a water displacement, and having third been used to displace for recovery previously produced spent cooking liquor from previously produced cellulosic pulp.

2. The process of claim 1 wherein the cellulosic fibrous material is cooked and the spent cooking liquor is displaced from the cellulosic pulp in contiguous zones within a hydraulic system, each zone being distinguished by conditions of temperature and alkali concentration.

3. The process of claim 1 wherein the cellulosic fibrous material is cooked, the spent cooking liquor is displaced from the cellulosic pulp, and the alkaline liquor is displaced by water from cellulosic pulp in contiguous zones within a hydraulic system, each zone being distinguished by conditions of temperature and alkali concentration.

4. A process of pulp manufacture, purification, and preparation for bleaching in which cellulosic fibrous material containing organic material including lignin and hemicelluloses is successively cooked at temperatures over 212° F. to produce cellulosic pulp and black liquor suitable for recovery, washed to displace for recovery the lignin and black liquor from the cellulosic pulp, and steeped at temperatures under 212° F. to dissolve the hemicellulose with an alkaline liquor which travels countercurrently to the cellulosic fibrous material in that it is successively displaced from steeped, previously produced cellulosic pulp with water, displaces previously produced black liquor from previously produced cellulosic pulp, and cooks cellulosic fibrous material.

5. A process of pulp manufacture, purification, and preparation for bleaching which comprises a first step of continuously cooking cellulosic fibrous material with an alkaline cooking liquor to produce cellulosic pulp and spent alkaline cooking liquor suitable for recovery, a second step of countercurrently displacing for recovery the spent alkaline cooking liquor with all of an alkaline liquor more concentrated in alkali than the spent alkaline cooking liquor and previously used to steep previously produced cellulosic pulp, a third step of countercurrently steeping the cellulosic pulp in said alkaline liquor more concentrated in alkali than the spent alkaline cooking liquor and fortified with make-up alkali, and a fourth step of countercurrently washing the steeped cellulosic pulp with water to obtain more of the alkaline liquor more concentrated in alkali than the spent alkaline cooking liquor.

6. A process which comprises performing the steps of cooking cellulosic fibrous material with alkaline liquor thereby producing pulp containing alkaline black liquor, then continuously and sequentially treating the pulp by displacing said black liquor with alkaline liquor which is more concentrated in alkali than said black liquor, steeping the pulp in said more concentrated alkaline liquor thereby dissolving hemicelluloses, displacing said more concentrated alkaline liquor from the pulp, and recirculating said more concentrated alkaline liquor which was displaced from the pulp, so as to make said more concentrated alkaline liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor and remove said hemicelluloses from said pulp and to use said more concentrated alkaline liquor over and over again for said sequential black liquor displacement and steeping.

7. A process which comprises performing the steps of cooking cellulosic fibrous material with alkaline liquor thereby producing pulp containing alkaline black liquor, then continuously and sequentially treating the pulp by displacing said black liquor from said pulp with part of an alkaline liquor which is more concentrated in alkali

than said black liquor, diluting said pulp with another part of said more concentrated alkaline liquor, steeping the pulp in said more concentrated alkaline liquor thereby dissolving hemicelluloses, displacing said more concentrated alkaline liquor and hemicelluloses from the pulp, and recirculating said more concentrated alkaline liquor which was displaced from the pulp, so as to make said more concentrated alkaline liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor and remove said hemicelluloses from said pulp and to use said more concentrated alkaline liquor over and over again for said sequential black liquor displacement and steeping.

8. A process which comprises performing the steps of cooking cellulosic fibrous material at a temperature above 212° F. and a pressure above atmospheric with alkaline liquor thereby producing pulp containing alkaline black liquor, then continuously and sequentially treating the pulp by displacing said black liquor from said pulp with alkaline liquor which is more concentrated in alkali than said black liquor, steeping said pulp in said more concentrated alkaline liquor at a temperature below 212° F., at substantially atmospheric pressure thereby dissolving hemicelluloses, displacing said more concentrated alkaline liquor and hemicelluloses from the pulp with water, and recirculating said more concentrated alkaline liquor which was displaced from the pulp, so as to make said more concentrated alkaline liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor and remove said hemicelluloses from said pulp and to use said more concentrated alkaline liquor over and over again for said sequential black liquor displacement and steeping.

9. A process which comprises performing the steps of cooking cellulosic fibrous material with alkaline liquor thereby producing pulp containing alkaline black liquor, then continuously and sequentially treating the pulp by displacing said black liquor from said pulp with alkaline liquor which is more concentrated in alkali than said black liquor, adding make-up alkaline liquor to said pulp, steeping the pulp in said more concentrated and make-up alkaline liquors thereby dissolving hemicelluloses, displacing the alkaline liquor and hemicelluloses from the pulp, and recirculating part of said last displaced liquor, so as to make said part of said last displaced liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor thereby also removing said hemicelluloses and to use said part of said last displaced liquor over and over again for said sequential black liquor displacement and steeping, and recirculating another part to cook said fibrous material.

10. A process which comprises performing the steps of cooking cellulosic fibrous material with alkaline liquor at a temperature above 212° F. and a pressure above atmospheric thereby producing pulp containing alkaline black liquor, then continuously and sequentially treating the pulp by displacing said black liquor from said pulp with alkaline liquor which is more concentrated in alkali than said black liquor, diluting said pulp with additional said more concentrated alkaline liquor and adding make-up liquor to bring the pulp to a consistency between 1% and 6% at an alkaline concentration between 40 and 150 grams per liter as apparent NaOH, steeping the pulp at said consistency and concentration at a temperature between 70° F. and 180° F. for a period of time between 5 and 120 minutes thereby dissolving hemicelluloses, displacing the alkaline liquor and hemicelluloses from the pulp and recirculating part of said last displaced liquor, so as to make said part of said last displaced liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor from said pulp and to use said part of said last displaced liquor over and over again for said sequential black liquor displacement and steeping and recirculating another part

to cook said cellulosic fibrous material thereby also removing said hemicellulose.

11. A process which comprises performing the steps of at least partially hydrolyzing cellulosic fibrous material at elevated temperature and pressure, cooking said material with alkaline sulfate liquor thereby producing pulp containing alkaline sulfate black liquor, then continuously and sequentially treating the pulp by displacing said alkaline sulfate black liquor from said pulp with alkaline sulfate white liquor which is more concentrated in alkali than said black liquor, adding make-up sulfate white liquor to said pulp, steeping the pulp in said more concentrated alkaline sulfate liquor thereby dissolving hemicelluloses, displacing said more concentrated alkaline sulfate liquor from the pulp with water, recirculating part of said more concentrated alkaline sulfate liquor which was displaced from the pulp, so as to make said part of said more concentrated alkaline sulfate liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor and remove said hemicelluloses from said pulp and to use said part of said more concentrated alkaline sulfate liquor over and over again for said sequential black liquor displacement and steeping, and recirculating another part of said more concentrated alkaline sulfate liquor for cooking said material.

12. A process which comprises performing the steps of cooking cellulosic fibrous material with alkaline liquor thereby producing pulp containing alkaline black liquor, fortifying a part of an alkaline liquor which is more concentrated in alkali than said black liquor with an alkaline solution containing up to about 75% by weight of an alkali, then continuously and sequentially treating the pulp by displacing said black liquor from pulp with said part of said more concentrated alkaline liquor, diluting said pulp with another part of said more concentrated alkaline liquor, steeping the pulp in said more concentrated alkaline liquor thereby dissolving hemicelluloses, displacing said more concentrated alkaline liquor and hemicelluloses from the pulp, and recirculating said more concentrated alkaline liquor which was displaced from the pulp, so as to make said more concentrated alkaline liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor and remove said hemicelluloses from said pulp and to use said more concentrated alkaline liquor over and over again for said sequential black liquor displacement and steeping.

13. A process which comprises performing the steps of cooking cellulosic fibrous material with alkaline liquor thereby producing pulp containing alkaline black liquor, then continuously and sequentially treating the pulp by displacing said black liquor from said pulp with part of an alkaline liquor which is more concentrated in alkaline than said black liquor, diluting said pulp with another part of said more concentrated alkaline liquor, steeping the pulp in said more concentrated alkaline liquor and in the presence of at least one surfactant thereby dissolving hemicelluloses, displacing said more concentrated alkaline liquor and hemicelluloses from the pulp, and recirculat-

ing said more concentrated alkaline liquor which was displaced from the pulp, so as to make said more concentrated alkaline liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor and remove said hemicelluloses from said pulp and to use said more concentrated alkaline liquor over and over again for said sequential black liquor displacement and steeping.

14. A process which comprises performing the steps of cooking cellulosic fibrous material with alkaline black liquor, then continuously and sequentially treating the pulp by displacing said black liquor from said pulp with part of an alkaline liquor which is more concentrated in alkali than said black liquor, diluting said pulp with another part of said more concentrated alkaline liquor, cooling the pulp in said more concentrated alkaline liquor, steeping the pulp in said more concentrated alkaline liquor thereby dissolving hemicelluloses, displacing said more concentrated alkaline liquor and hemicelluloses from the pulp, and recirculating said more concentrated alkaline liquor which was displaced from the pulp, so as to make said more concentrated alkaline liquor travel from near the terminal portion of the process to the initial area of treatment to displace said black liquor and remove said hemicelluloses from said pulp and to use said more concentrated alkaline liquor over and over again for said sequential black liquor displacement and steeping.

15. A process which comprises performing the steps of cooking cellulosic fibrous material with alkaline liquor thereby producing pulp containing alkaline black liquor, then continuously and sequentially treating the pulp by displacing said black liquor from said pulp with part of an alkaline liquor which is more concentrated in alkali than said black liquor, diluting said pulp with another part of said more concentrated alkaline liquor, steeping the pulp in said more concentrated alkaline liquor thereby dissolving hemicelluloses, displacing said more concentrated alkaline liquor and hemicelluloses from the pulp, recirculating said more concentrated alkaline liquor which was displaced from the pulp, so as to make said more concentrated alkaline liquor travel from near the terminal portion of the process back to the initial area of treatment to displace said black liquor and remove said hemicelluloses and to use said more concentrated alkaline liquor over and over again for said sequential black liquor displacement and steeping, acidifying the pulp, washing the pulp, neutralizing the pulp, and bleaching the pulp first with chlorine dioxide and then with at least one hypochlorite.

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