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PRODUCTION OF CELLULOSE PULP

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The present invention relates to a process for making pulp and more particularly, relates to a process for making pulp from cellulosic materials in which the cellulosic material is subjected to pretreatment which greatly enhances the rapidity and uniformity with which such material may be penetrated by a cooking liquid. It has long been recognized that rapid and uniform penetration leads to the production of uniformly cooked pulp with attendant advantages from the standpoint of yield and quality. It also has been recognized that air within the interstices or canals of the cellulosic material is one of the factors which greatly affects the rate and uniformity of penetration.

In the common chemical pulping of wood tissue in the form of chips, or of other cellulosic material, it is customary to subject said material to the action of a chemical pulping solution in a pressure vessel according to a predetermined time, temperature, pressure cycle after which the pulped tissue is discharged under pressure or after the relief of pressure, from the vessel, as may be desired. Furthermore, it is sometimes the custom to subject the cellulosic charge in the cooking vessel or in a bin or hopper communicating with the cooking vessel, to a steaming operation at or near atmospheric pressure prior to the introduction of the cooking liquor. Such steaming serves to displace the air surrounding the subdivided material (such as chips), and possibly some of the air within the subdivided material with steam or a mixture of air and steam. This serves to improve penetration. A much greater improvement in penetration is achieved by the process disclosed in co-pending United States application Serial No. 105,162 filed July 16, 1949, by Maass, Ross, Hart and Strapp, now Patent No. 2,640,774, granted June 2, 1953 and the co-pending Canadian application Serial No. 590,662 filed July 28, 1949, wherein the subdivided material is subjected, after steaming at atmospheric pressure, to a further steaming at a pressure above atmospheric after which such higher pressure is substantially and rapidly decreased by the removal or withdrawal of the air-steam mixture from the interior of the cellulosic material and from the pressure vessel without the reintroduction of air into the vessel prior to the introduction of the cooking liquor. In the latter process the withdrawal of the mixture of air and steam from the interior of the subdivided material conditions it for rapid and uniform penetration by an aqueous cooking liquid.

In the present invention cellulosic tissue, in the form of chips, for example, may be first steamed at or near atmospheric pressure with the material vented to the air to displace what air may be displaced from in and around it. Without intervening exposure to additional air the material is then steamed at a super-atmospheric pressure and submerged in a chemical pulping liquor under such conditions as to maintain the same super-atmospheric pressure within the system. Under these conditions the chips will be penetrated with sufficient chemical pulping liquor, and therefore chemical, to effect

rapid and uniform pulping. The process of the present invention may be carried out in batch or continuous apparatus and in some instances the preliminary steaming at or near atmospheric pressure may be omitted.

In a general sense the distinguishing feature of the present invention is my discovery that extremely rapid and uniform penetration may thus be achieved in spite of the fact that the actual weight of air within the interstices or canals of the subdivided material may not be substantially reduced by the steaming operation at super-atmospheric pressure, provided that the subdivided material is submerged in the cooking liquor under such conditions that there is no substantial loss of pressure within the pressure vessel in which the material has been steamed at super-atmospheric pressure and in which the submersion of the material occurs.

In order to prevent substantial loss of said super-atmopsheric pressure during the submersion step it is necessary to introduce the cooking liquor under a maintained pressure at least equal to said super-atmospheric pressure. Also it is necessary to add it at such a rate that the increasing volume of liquor will at least compensate for such condensation or dissolution of the steam into the incoming liquor as may occur. If the cooking liquor is preheated to a temperature approaching, but definitely below, the boiling point of water the rapidity of condensation will be reduced whereas if it is relatively cool when introduced the rapidity of condensation will be increased. Thus cooking liquor at, say, 50° C. must be introduced at a faster rate than cooking liquor at, say 90° C. However, more steam may be introduced along with the cooking liquor if it is not desirable or practical to introduce the liquor at a rate sufficiently rapid to maintain the pressure without the addition of more steam. The maintained super-atmospheric pressure, however, preferably should not be achieved by heating the liquor to a point where its aqueous vapour pressure is substantially equal to or above that of the super-atmospheric pressure since it is believed that under such conditions the advancement of liquor into the cellulosic material may be retarded. From another standpoint it is not desirable to preheat the cooking liquor to the temperature at which it will react rapidly with the cellulosic material since at such temperature the material would start to cook before penetration was complete with resultant danger of overcooking of exterior fibers or undercooking of interior fibers. Thus for ordinary aqueous cooking liquor the temperature of the liquor at time of addition to the cellulosic material should lie within a range of from about 40° C. to about 90° C., although this is a practical, rather than a critical, range as will be readily apparent.

If it is desired to avoid further increase in pressure within the vessel during introduction of the cooking liquor, the vessel may be vented to permit escape of steam but in no event should the pressure within the vessel be permitted to drop substantially below the pressure established during the super-atmospheric steaming.

While I do not intend that this invention be limited by reference to any particular theory, it appears that the steaming of subdivided cellulosic tissue at super-atmospheric pressure will result in the air within such tissue being compressed and mixed with the steam. At the same time the air adsorbed on the walls of the interstices will be replaced in some proportion by water molecules, possibly to the extent of an actual liquid layer. The walls of the interstices are then in such a partially wetted condition that when the cooking liquor is introduced it can, in turn, wet these walls and the chemical which it carries will immediately diffuse into them. Since the air is compressed and the walls are wetted, the air cannot serve as a serious impedence to the penetration by the cooking liq-

uor as appears to be the case when the cooking liquor is introduced under pressure without previous steaming at super-atmospheric pressures. Whether this theory is wholly or even partially correct, the quality of pulp produced by my process gives clear and unmistakable evidence of rapid and thorough penetration since the resulting pulp is unusually uniform as regards the absence of overcooked or undercooked portions.

The value of such results is important in both batch and continuous pulping operations and the process herein described is clearly applicable to both operations. Beyond this the process is admirably suited to continuous operation because, except for the initial steaming at atmospheric pressure, the entire penetration pretreatment and pulping operation may be carried out at the single super-atmospheric pressure selected as best suited for the particular raw cellulosic material, the particular cooking conditions to be used and the particular type of product desired. The apparatus required may thus be much simpler in construction and operation than that used where several different super-atmospheric pressures are necessary.

Specific examples of batch and continuous pulping operations embodying the present invention will now be given.

Example 1.—Wood chips are placed in a digester suitable for batch operation. The chips are steamed with a vent open to the air in order to displace the air surrounding the chips and perhaps some of the air from within the chips. When the flow of steam from the vent indicates that substantially complete displacement has been reached, the vent may be closed and the steam pressure is then raised to 100 lbs. per square inch. In the meantime an aqueous cooking liquor of any desired type is heated in a separate container to a temperature of approximately 90° C. For the purposes of the present example, the cooking liquor may comprise a caustic soda solution of the type ordinarily used in the soda process. The cooking liquor is then subjected to a pressure of a few pounds more than 100 lbs. per squar inch. Such pressure may be established by admitting steam to the top of the container for the cooking liquor. The liquor is then transferred under such pressure, which may be maintained by a continued admission of steam, to the digester. Preferably the cooking liquor is introduced at the bottom of the digester to reduce turbulence. As the liquid level rises in the digester the increasing volume of liquid will tend to maintain the gaseous pressure therein at a high level. The liquor thus should be supplied to the digester at such a rate as at least to maintain the gaseous pressure at 100 lbs. per square inch. If the rate of addition is sufficiently rapid to cause increase in gaseous pressure above 100 lbs. per square inch and to an undesired level, the digester may be carefully vented so long as the pressure is not permitted to drop substantially below 100 lbs. per square inch. When all of the chips have been submerged in liquor, it is permitted to remain in contact with the chips for about ten minutes under the pressure of 100 lbs. per square inch and thereafter steam is applied directly to the digester, and the liquor is drained from the chips in such manner that steam replaces the surrounding liquor at a pressure of 100 lbs. per square inch. When the surrounding liquor is removed the cooking reaction is continued in the absence of liquor other than that previously imbibed by the chips. With the soda-cook liquor of the present example this phase of the operation is continued at 100 lbs. per square inch steam pressure for 30 minutes. The cooked pulp is then removed from the digester. The pulp resulting from the process of the present example is uniformly cooked pulp substantially free of undercooked or overcooked portions.

Example 2.—Wood chips are supplied to a continuous digester preferably of the type having several successive horizontally disposed pipes through which the cellulosic material is continuously moved. In some instances the chips are introduced to such a digester from a hopper in which they may be steamed at or near atmospheric pressure so that the air surrounding the chips and perhaps some of the air within the chips will be displaced by steam before the chips actually enter the first horizontal pipe of the digester. In such instances the super-atmospheric steaming step of the present invention may be carried out in the first horizontal pipe. In other types of continuous digesters it may be necessary to employ the first horizontal pipe or a section or zone thereof for the atmospheric steaming step in which event the first horizontal pipe will be provided with vents to the atmosphere and suitable inlets for steam to assure displacement of air during the passage of the chips through such first horizontal pipe. In either event the horizontal pipe to which the chips are introduced after the atmospheric steaming step is provided with a suitable inlet valve or other pressure maintaining device or provision by which relatively high pressure may be maintained upon the chips without danger of them blowing back into the lower pressure region wherein the atmospheric steaming occurred. After the chips enter the high pressure zone of the continuous digester, steam is admitted at one or more points to raise the pressure upon the chips to the desired point. For the purposes of the present example the steam pressure may be raised to 100 lbs. per square inch in a zone through which the chips are progressively advanced toward the end of the particular horizontal pipe. The chips under such pressure may then be advanced into the next section or horizontal pipe of the digester wherein a bath of cooking liquid is maintained under the same pressure as exists in the superatmospheric steaming section or pipe. The chips thus progress from an atmophere of steam at 100 lbs. per square inch into the bath of cooking liquor without any loss of pressure. In this section or pipe of the digester the chips imbibe the cooking liquor which will penetrate them rapidly and uniformly after which they may enter the next section or pipe of the digester. Preferably the cooking liquor is separated from the chips as they move into the next pipe and to this end an appropriate circulating and makeup system will be provided for the section or pipe of the digester in which submersion occurs. The chips separated from the immersion bath are cooked in the absence of surrounding liquor in the next pipe by maintaining a steam pressure of 100 lbs. per square inch therein and the finished pulp is discharged at the end of this last section or pipe of the continuous digester.

Since the gaseous pressure maintained in the super-atmospheric steaming section or pipe and all of the succeeding sections or pipes in which immersion and cooking occurs, it is not necessary to provide valves or equivalent structure for separating these pipes or sections into different pressure zones. The process of the present example thus affords the advantages of uniform penetration in a continuous digesting system of extremely simple construction.

In either of Examples 1 or 2 herein, it will be understood that the particular pressure and temperature relationships may be varied in accordance with the nature of the raw cellulosic material, the particular cooking liquor employed and the pulp characteristics desired. Thus higher tempertures and pressures will bring about more rapid penetration and cooking and lower temperatures and pressures will bring about slower penetration and cooking. Variations in temperature, pressure and time may thus be resorted to to produce pulps of various uniform qualities from widely varying sources of cellulosic fiber. The essential advantage of the present invention is the rapid and uniform penetration which it affords and such advantage may be availed of to produce a higher quality of pulp in a given time or to produce a particular quality of pulp in a shorter time as will be well understood by those skilled in the art.

The pressure used in the super-atmospheric steaming step and which is maintained during immersion of the raw material in cooking liquor may lie substantially any-

where within the range of pressures ordinarily used in the pulping of cellulosic raw material and in some instances may be higher than these usual ranges if so desired. The 100 lbs. per square inch gauge pressure selected for Examples 1 and 2 is merely illustrative of a useful pressure. In normal batch operation for the production of various kinds of cellulosic pulp from various kinds of raw material the maximum pressure reached in cooking is at least about two atmospheres and will usually lie within a range of gauge pressures of from about 50 lbs. per square inch which might be used for indirect sulphite cooking, to about 120 lbs. per square inch, the higher pressures being frequently used in soda or sulphate cooking. In continuous operations the range of pressures may lie between 50 lbs. per square inch and 250 lbs. per square inch when presently available commercial equipment is used. The improved penetration achieved by my invention is readily apparent within any of the ranges of pressure thus likely to be encountered in adapting the same to commercial procedure. Furthermore, it is within the purview of the present invention to steam the raw material and to impregnate it at pressure higher than those which may be used in the subsequent cooking steps.

One particular advantage of the present invention lies in the fact that the raw material is so completely and uniformly penetrated by the cooking liquor that it is possible to separate the unabsorbed cooking liquor from the chips and to complete the cook in the absence of this unabsorbed liquor as set forth in Examples 1 and 2. It will be apparent, however, that the present invention is not limited to processes in which such separation occurs and that it may be adapted to processes in which final cooking is carried out with the raw material either partially or wholly submerged in the cooking liquor.

I claim:

1. In the production of cellulosic pulp the process of impregnating a finely divided cellulosic material with an aqueous cooking liquor comprising placing said cellulosic material in a pressure vessel, introducing steam to said pressure vessel to establish an atmosphere of steam and air in direct contact with said cellulosic material at a super-atmospheric pressure of at least about two atmospheres before the introduction of cooking liquor to said cellulosic material, and thereafter forcing an aqueous cooking liquor into said pressure vessel to submerge said cellulosic material while maintaining said cellulosic material and the liquor in which it is being submerged under a pressure at least as great as said super-atmospheric pressure.

2. In the production of cellulosic pulp the process of impregnating a finely divided cellulosic material with an aqueous cooking liquor comprising steaming said cellulosic material before the introduction of cooking liquor thereto and while it is vented to the air to establish an atmosphere ambient to said cellulosic material which consists principally of steam, raising the pressure of said ambient steam atmosphere to a super-atmospheric value of at least about two atmospheres, and thereafter submerging said cellulosic material in an aqueous cooking liquor while maintaining said cellulosic material and the liquor in which it is being submerged under a pressure at least substantially as great as said super-atmospheric pressure.

3. In the production of cellulosic pulp the process of impregnating a finely divided cellulosic material with an aqueous cooking liquor comprising placing said cellulosic material in a pressure vessel, introducing steam to said pressure vessel to establish an atmosphere of steam and air in direct contact with said cellulosic material at a super-atmospheric pressure of at least about two atmospheres before the introduction of cooking liquor to said cellulosic material, and thereafter forcing an aqueous cooking liquor into said pressure vessel to submerge said cellulosic material while maintaining substantially constant the super-atmospheric pressure within said pressure vessel.

4. In the production of cellulosic pulp the process of impregnating a finely divided cellulosic material with an aqueous cooking liquor comprising steaming said cellulosic material before the introduction of cooking liquor thereto and while it is vented to the air to establish an atmosphere ambient to said cellulosic material which consists principally of steam, raising the pressure of said ambient steam atmosphere to a super-atmospheric value of at least about two atmospheres, and thereafter submerging said cellulosic material in an aqueous cooking liquor while maintaining upon said cellulosic material and the liquor in which it is being submerged a substantially constant pressure equal to said super-atmospheric pressure.

5. A process for the production of cellulosic pulp from a finely divided cellulosic material comprising placing said cellulosic material in a pressure vessel, introducing steam to said pressure vessel to establish an atmosphere of steam and air in direct contact with said cellulosic material at a super-atmospheric pressure corresponding to the total pressure of the cooking liquor to be used at a temperature appropriate for chemical pulping of said cellulosic material therein, thereafter forcing an aqueous cooking liquor into said pressure vessel to submerge and impregnate said cellulosic material while maintaining said cellulosic material and the liquor in which it is being submerged under a pressure at least substantially as great as said super-atmospheric pressure, and thereafter cooking said impregnated cellulosic material.

6. A process for the production of cellulosic pulp from a finely divided cellulosic material comprising steaming said cellulosic material before the introduction of cooking liquor thereto and while it is vented to the air to establish an atmosphere ambient to said cellulosic material which consists principally of steam, raising the pressure of said ambient steam atmosphere to a super-atmospheric value corresponding to the total pressure of the cooking liquor to be used at a temperature appropriate for the chemical pulping of said cellulosic material therein, submerging said cellulosic material in an aqueous cooking liquor while maintaining said cellulosic material and the liquor in which it is being submerged under a pressure at least substantially as great as said super-atmospheric pressure to impregnate said cellulosic material with said cooking liquor and thereafter cooking said impregnated cellulosic material.

7. A process for the production of cellulosic pulp from a finely divided cellulosic material comprising placing said cellulosic material in a pressure vessel, introducing steam to said pressure vessel to establish an atmosphere of steam and air in direct contact with said cellulosic material at a super-atmospheric pressure corresponding to the total pressure of the cooking liquor to be used at a temperature appropriate for chemical pulping of said cellulosic material therein, thereafter forcing an aqueous cooking liquor into said pressure vessel to submerge and impregnate said cellulosic material while maintaining substantially constant the super-atmospheric pressure within said pressure vessel, and thereafter cooking said impregnated cellulosic material while continuing to maintain substantially constant the super-atmospheric pressure within said pressure vessel.

8. A process for the production of cellulosic pulp from a finely divided cellulosic material comprising steaming said cellulosic material before the introduction of cooking liquor thereto and while it is vented to the air to establish an atmosphere ambient to said cellulosic material which consists principally of steam, raising the pressure of said ambient steam atmosphere to a super-atmospheric value corresponding to the total pressure of the cooking liquor to be used at a temperature appropriate for chemical pulping of said cellulosic material therein, submerging said cellulosic material in an aqueous cook-

ing liquor while maintaining upon said cellulosic material and the liquor in which it is being submerged a substantially constant pressure equal to said super-atmospheric pressure, to impregnate said cellulosic material with cooking liquor and thereafter cooking said impregnated cellulosic material while continuing to maintain said cellulosic material under a pressure substantially equal to said super-atmospheric pressure.

9. A process as set forth in claim 5 wherein after said cellulosic material has been impregnated and before it is cooked, it is separated from the cooking liquor in which it was submerged while continuing to maintain said impregnated cellulosic material under a pressure at least substantially as great as said super-atmospheric pressure.

10. A process as set forth in claim 6 wherein after said cellulosic material has been impregnated and before it is cooked, it is separated from the cooking liquor in which it was submerged while continuing to maintain said impregnated cellulosic material under a pressure at least substantially as great as said super-atmospheric pressure.

11. A process as set forth in claim 7 wherein after said cellulosic material has been impregnated and before it is cooked, it is separated from the cooking liquor in which it was submerged while continuing to maintain substantially constant the super-atmospheric pressure within said pressure vessel.

12. A process as set forth in claim 8 wherein after said cellulosic material has been impregnated and before it is cooked, it is separated from the cooking liquor in which it was submerged while continuing to maintain substantially constant the super-atmospheric pressure upon said impregnated cellulosic material.

13. A continuous process for the production of cellulosic pulp from a finely divided cellulosic material comprising feeding said cellulosic material progressively through a continuous pulp-digesting apparatus, supplying steam to said apparatus to establish an atmosphere of steam and air in direct contact with said cellulosic material before it is brought into contact with cooking liquor and under a super-atmospheric pressure corresponding to the total pressure of the cooking liquor to be used at a temperature appropriate for chemical pulping of said cellulosic material therein, supplying cooking liquor to said apparatus at a hydrostatic pressure at least slightly higher than said super-atmospheric pressure while maintaining said super-atmospheric pressure substantially constant to impregnate said cellulosic material with cooking liquor, adding heat to said apparatus to cook said impregated cellulosic material while continuing to maintain said super-atmospheric pressure substantially constant, and continuously discharging the cooked cellulosic material from said apparatus.

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