

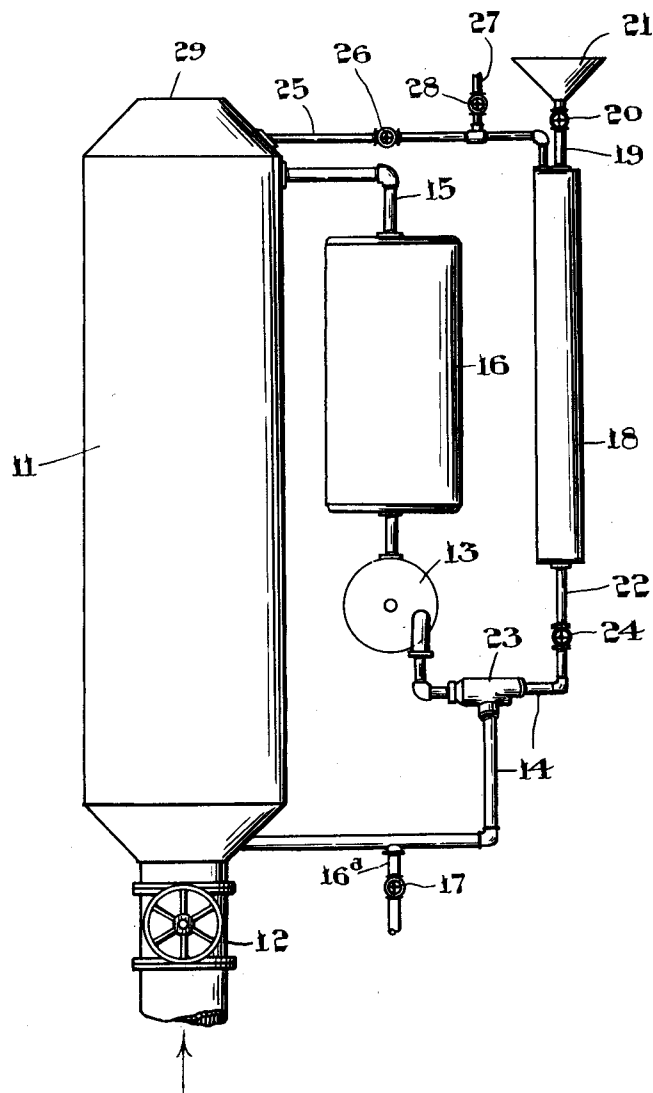
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PROCESS OF MAKING CHEMICAL PULP

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PROCESS OF MAKING CHEMICAL PULP

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This invention relates to the manufacture of cellulose or chemical pulp from wood or other fibrous materials. More particularly, the invention is chiefly concerned with the digesting of the raw materials and comprises a method and means whereby the character or concentration of the active reagents in contact with the fibrous materials at any stage of the digesting process may be selectively controlled to maintain conditions necessary for producing pulp of certain improved characteristics. The aforesaid concentration of the active reagents may, in accordance with this invention, be increased or decreased at any desired stage or may be maintained at a substantially constant predetermined value throughout the whole or any part of the digesting process. Likewise, the character of the active reagent in contact with the fibrous materials at any stage of operation is also subject to selective control.

In ordinary commercial practice the chipped wood or other raw material to be pulped is charged into a digester; covered with a solution of the active reagent; and heated until the undesirable constituents of the material are rendered soluble, thus leaving the cellulose pulp as a residue. This process of digesting the raw material is commonly referred to as "cooking". The character and concentration of the reagent, the temperature at which the "cooking" is carried out, and the time required to complete the "cook" are subject to considerable variation depending, largely, upon the nature of the raw material and the character of pulp to be produced. The correlation and control of these various factors constitutes the technique of commercial cellulose or chemical pulp manufacture.

The pulping processes in common use today such, for example, as the soda, sulphate, keeba, and sulphite processes do not contemplate the exercise of any selective control with reference to the character or concentration of the active reagents in contact with the fibrous material throughout the various stages of the cooking cycle. I have discovered, however, that such control is of prime importance in that it removes certain limitations now governing the carrying out of these processes and enables the production of pulp having certain desirable specific characteristics impossible to obtain under present conditions from the same raw materials.

In all of the processes referred to, it is necessary at the commencement of the "cook" to place in the reaction vessel a cooking liquor containing an active reagent in amounts sufficient to last

throughout the entire course of the "cook". This naturally requires an excessively high initial concentration which, at the temperatures employed in commercial practice, is known to have a degrading action on the cellulose of the fibrous material. This degradation is shown by the viscosity test and the alpha cellulose test. Such tests indicate a specific characteristic of the fibre such as the "micelle" size of the molecular aggregates of the cellulose. For certain purposes it is essential that the "micelle" size be large and this is particularly true if the pulp produced is to be used in connection with the manufacture of artificial silk.

Take, for example, the soda process. In this process, the amount of active alkali contained in the cooking liquor which is placed in the reaction vessel at the commencement of the cooking cycle must be such as to assure a sufficiency of the active reagent for pulping throughout the entire course of the "cook". In ordinary commercial practice, this requires the use of alkaline solutions such as caustic soda, having an initial concentration of 6 to 7%. The amount of alkali in the solution usually ranges from 20 to 25% of the weight of the wood contained in the reaction vessel. During the course of the "cook" about two thirds to three fourths of this alkali combines with wood constituents so that the final concentration is only about one fourth of the initial concentration. Hence, in the soda process, in common with all other chemical processes, the required initial concentration of the active reagents is necessarily at such a high value as to have the aforementioned degrading action on the cellulose and is known to produce a definite reduction of the "micelle" size as shown by low viscosity and low alpha cellulose. There is, therefore, a condition in the carrying out of the soda process which inhibits the production of a pulp having the "micelle" size and other characteristics best suited for the manufacture of artificial silk and for other purposes for which pulp produced in accordance with the present invention may be successfully used.

The direct advantage of the method of control afforded by this invention whether embodied in the soda or other chemical pulping processes now in use, is that it renders possible a better control of reaction conditions detrimental to the quality of the pulp while maintaining the conditions necessary for isolation and purification of the fibre. In other words, the character and/or concentration of the cooking liquor is subject to such selective control throughout the cooking cycle as

to be at all times of the character or concentration most appropriate for the establishment of the optimum pulping conditions for the stage of cooking then in progress.

The concentration may be controlled in any suitable manner by the periodic or continuous addition of a suitable solution of the cooking liquor at a concentration different from that already contained in the reaction vessel. In practice the particular concentration to be established or maintained for any stage of the "cook" will, of course, vary in accordance with the cooking liquor in use; the character of the wood or other fibrous material; the extent to which the cooking has been carried out, and the characteristics desired in the final pulp. The character of the cooking liquor in contact with the fibrous material is subject to selective control in the sense that, at any state of operation, a cooking liquor of characteristics different from that then contained in the reaction vessel may be introduced whenever it is necessary or desirable to promote reaction conditions that would not otherwise occur.

In carrying out the soda process, for example, with the aid of the control afforded by this invention, it is possible to commence the "cook" with a cooking liquor having a relatively low initial concentration of about 2% and by successive or continuous addition of a small amount of a suitably concentrated solution of alkali to maintain the initial concentration of 2% of active alkali throughout the "cook". Such addition of chemical reagents may be accomplished either manually or automatically by any suitable means. A suggested procedure is the introduction of the said chemicals into an external circulating or heating circuit forming part of the digester installation. The reagents added at any state of operation will usually be of a concentration different from that of the active reagent then in contact with the fibrous materials.

In order to compensate for the added volume of chemical reagents it may, in some instances, be necessary to eliminate an equivalent volume of exhausted or partly exhausted liquor from the reaction vessel. This may be accomplished by providing the reaction vessel with a relief line capable of manual, semi-automatic, or automatic manipulation. This relief line may also be used for extracting portions of the cooking liquor for testing purposes so that the concentration of the chemical reagents in the cooking liquor may be conveniently determined and the addition of fresh reagents signified for introduction into the system to maintain the desired conditions.

In carrying out the soda process in accordance with this invention, it is also advantageous at times to commence the cook with a very weak solution of alkali or with plain water. The wood or other material is heated for a suitable period of time in contact with this starting solution and alkali is then added to raise the concentration to the desired value which is therefore subject to selective variation or may be maintained, constant throughout the remainder of the cook. The concentration employed, after heating with the starting solution may be 2% or any other desired concentration. The end point of the cooking operation may be decided by the amount of alkali actually combined with the wood as measured by tests of the amount and concentration of the cooking liquor.

While the feature of establishing and maintaining a relatively low and substantially con-

stant concentration throughout the whole or major portion of the cook is of important advantage for certain conditions of operation, this invention also contemplates a much wider range of control. For instance, the concentration of active reagents in contact with the material at one stage of operation may be reduced for a succeeding stage of operation by dilution or by replacing the concentration then in use either in whole or in part by a solution of lower concentration. It is therefore apparent that both the character and the concentration of the cooking liquor in contact with the fibrous material at any given stage is subject to a wide range of control to facilitate the establishment and maintenance of the most favourable pulping conditions throughout the digesting process.

In the foregoing discussion, the invention has been described particularly with reference to the carrying out of the soda process, but it will be apparent that similar control and advantages obtain when the invention is embodied in the sulphite or other chemical pulping processes now in use.

In the further discussion of this invention reference will be had to the single figure of the accompanying drawing wherein I have shown a diagrammatic illustration of a simple form of apparatus suitable for carrying the invention into effect.

Referring more particularly to the drawing, 11 designates a conventional form of digester which is equipped with the usual bottom inlet 12 for the introduction of steam. During the digesting process the cooking liquor is continuously circulated from the bottom to the top of the digester or vice versa through an external circulating and/or heating circuit. This circuit comprises a pump 13 having its inlet connected to the bottom of the digester by a pipe 14 and its outlet connected to the top of the digester by a pipe 15. When external heating is being used a portion of the pipe 15 passes through a heat interchanger 16 of any suitable construction wherein the circulating liquor is heated prior to being returned to the top of the digester. The pipe 14 is provided with a relief line 16a controlled by a valve 17. By opening the valve 17 a portion of the cooking liquor may be withdrawn from the system for testing purposes or to make room for the liquor or reagents to be added for the purpose of controlling the concentration of the active reagents in contact with the fibrous material contained in the digester.

The added reagents or cooking liquor may be conveniently introduced into the system in the following manner. The reserve solution is initially introduced in a supply tank 18 through the medium of a supply line 19 equipped with a valve 20 and a filling funnel 21. From the tank 18 the reserve supply is conducted through a discharge pipe 22 leading to an injector 23 located in the circulating pipe 14 and from thence the reserve supply is drawn into the pump 13 for delivery to the digester. The discharge pipe 22 is controlled by a valve 24 which may be operated in any suitable manner.

Pressure may be supplied to assist in feeding the reserve supply from the tank 18 to the pump 13. For example, a portion of the gas accumulating in the upper portion of the digester may be conducted to the upper portion of the reserve supply tank 18, through a pipe 25 equipped with a control valve 26. In this way the pressure ex-

erted on the reserve supply contained in the tank 18 may be adjusted to equalize the pressure of the circulating circuit into which the reserve supply is introduced during the digesting process.

5 The same result may, however, be obtained by the use of steam supplied through a pipe 27 controlled by a valve 28. The pipe 27 is connected to the pipe 25 between the valve 26 and the reserve supply tank 18. As previously explained the character or concentration of the solution or active reagents supplied to the digester from the tank 18 will be predetermined to promote the desired reaction conditions without creating conditions detrimental to the quality of the pulp.

15 Complete data pertaining to a typical "cook" conducted in accordance with this invention is given in the appended table, which shows, among other things, the quantity of active reagent (sodium hydroxide) added to the fibrous material, the timing of such additions, and the concentration of active alkali then in contact with the fibrous materials. This table also indicates the yield and other characteristics of the "cook" conducted as prescribed therein.

Example cook

Digester used—special design—injector type.

Charge: (A) 3000 grams poplar chips (bone dry weight)

(B) 20000 grams water.

	Time	Temp. °C.	Liquor Conc. % NaOH.	Grams NaOH injected.
35	9.10 A.M.	25	0	
	9.42	100	0	569.5 (18 min.)
	10.08		1.34	
	10.10	138		279.3 (12 min.)
	10.32	145	1.95	
	11.10	170	1.65	
	11.19	170		74.1 (6 min.)
40	11.30	172	1.89	
	11.55	170	1.83	28.5 (10 min.)
	12.24 P.M.	170	2.01	51.3 (5 min.)
	12.45	170	2.01	
	3.45	170		
	4.42	140	(Charge blown from di- gester)	
45				

Per cent

Yield of screened pulp bone dry----- 53.25

Yield of screenings----- 1.48

50 Easy bleach test 100% easy bleach.

Lignin in Pulp 1.43% (Roe Chlorine Number 1.15)

Viscosity unbleached 36.38 c.p.

Minutes beating

	20	40	60	80	100	120
Basis wt. 25×40×500..	20	40	60	80	100	120
Bursting strength.....	53.5	77.2	87.3	95.3	101.7	111.6
Tearing strength.....	70	62	61	65	74	76
Metres breaking length	5740	6670	7400	8870	9330	9100

In carrying out this particular cook, the reserve or measuring covering tank 18 is filled with a strong liquor of approximately 20% NaOH weight. The wood or other fibrous material is charged into the digester through the top opening 29. A suitable quantity of water is added in the same manner and the opening 29 then sealed by the application of the usual closure (not shown). A heating medium is supplied to the heat interchanger 16 and the pump 13 is set in operation to commence the circulation. The strong solution contained in the tank 18 is fed through the injector 23 at such a rate as to mix evenly with the liquor circulated by the pump 13. The temperature is raised according to the definite time schedule shown on the appended table to a maximum of 170° and the concentration of the active reagent is controlled and predetermined by periodically extracting samples via the relief line 16a and injecting fresh liquor of the necessary concentration. The first sample is usually taken a few minutes after the final addition of the strong liquor supplied from the tank 18. At the conclusion of a predetermined cooking period the fibrous material is blown from the digester and subjected to the usual subsequent treatment such as washing, screening, etc. It will be noted that high liquor ratios were used in the sample cook described but this is not important since the control of concentration afforded by this invention practically eliminates liquor ratio as a factor affecting the final results.

Having thus described my invention what I claim is:—

1. In the manufacture of chemical pulp the step of maintaining the optimum pulping conditions throughout the course of the digesting process by selective control of the active reagents in contact with the fibrous material, which comprises withdrawing a portion at least of the digesting liquid, adding an active agent thereto in predetermined concentration, and returning the treated portion of the digesting liquid to the digesting process.

2. In the manufacture of chemical pulp by digestion of fibrous material by the action of chemicals in solution, the steps of continuously withdrawing liquid from the digesting solution, adding chemical to said liquid to maintain concentration at a predetermined point, and returning the chemically modified liquid to the digesting process.

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