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[54] **VAPOR PHASE PULPING OF WATER SATURATED LIGNOCELLULOSIC MATERIALS**

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[56]

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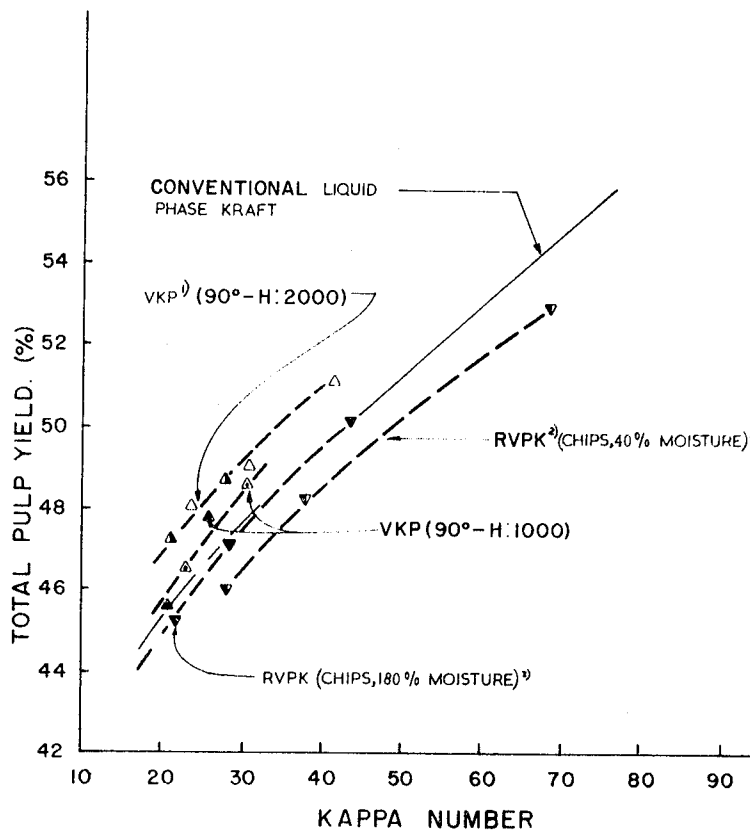
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ABSTRACT

Impregnating a water-saturated lignocellulosic material with an alkaline cooking liquor at a temperature below about 110° C., removing excess cooling liquor from the impregnated material, and delignifying the impregnated material by rapidly heating with steam.

6 Claims, 1 Drawing Figure



- 1) VKP = VAPOUR PHASE KRAFT PROCESS OF THE PRESENT INVENTION
 2) RVPK = RAPID VAPOUR PHASE KRAFT PROCESS AS TAUGHT BY
 KLEINART, U.S. PATENT 3,215,588
 3) MOISTURE CONTENTS BASED ON OVERDRY WEIGHT OF WOOD

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VAPOR PHASE PULPING OF WATER SATURATED LIGNOCELLULOSIC MATERIALS

The present invention relates to the production of cellulosic pulp from a lignocellulosic material by alkaline pulping, particularly kraft pulping, and in particular relates to an improved vapor phase alkaline pulping process for the production of a chemical cellulosic pulp in which the quality of the pulp and the yield of pulp is improved over the known vapor phase alkaline pulping process.

In the conventional liquid phase alkaline cooking process, in particular the kraft or sulphate process for the production of a chemical cellulosic pulp, in particular wood pulp, the subdivided wood or other lignocellulosic material is cooked in an aqueous solution of sodium hydroxide and sodium sulphide at a temperature of the order of 170° C. for the time required to produce the pulp in the required yield. The resulting pulp contains lignin and carbohydrates in a ratio which is determined by the specific conditions of the pulping process i.e. the temperature cycle, the time, the liquor-to-wood ratio and the ratio of chemical to wood. In general the ratio of carbohydrate to lignin in the pulp from a given material at a given yield is a quantity which varies within very narrow limits, being virtually fixed by the nature of the process.

In an attempt to improve on the conventional kraft process in which the wood chips are slowly cooked in an aqueous solution at 170° C., it is proposed by Kleinert inter alia in U.S. Pat. No. 3,215,588 issued Nov. 2, 1965 to effect the delignification of the wood chips by a rapid high temperature vapor phase cooking process upon chips previously impregnated with the kraft cooking liquor, by subjecting said chips to the action of steam, which is capable of rapidly raising the temperature of the chips to the cooking temperature which is in the range 170° to 185° C. Subsequently, the delignified chips are quenched in an aqueous processing liquor, such as water or dilute black liquor, which, besides rapidly cooling the chips, also facilitates their removal from the digester. As set forth by Kleinert in U.S. Pat. No. 3,215,588 it is possible to produce pulps of lower lignin content than are obtainable with the conventional liquid phase process, in which there is a slow rise in temperature.

In particular in the Kleinert process, normal dry wood chips are impregnated with the kraft cooking liquor at a temperature not exceeding 150° C., the excess of liquor is removed from the impregnated chips and the temperature of the chips is then rapidly raised to a high value in the range of 170° to 185° C. by steaming under pressure to effect delignification of the chips. The delignified chips are then quenched in an aqueous processing liquor.

According to the teaching of Kleinert with respect to his alkaline vapor phase pulping process in U.S. Pat. No. 3,215,588 and also in a series of papers on the same rapid vapor phase alkaline pulping process, entitled "Mechanisms of Alkaline Delignification", Tappi 49, No. 7, pages 301-303 (July, 1966); Tappi 49, No. 2, pages 53-57 (February, 1966) and Tappi 51, No. 10, pages 467-470 (October, 1968) as well as papers by Kleinert and Marraccini, Tappi 48, No. 3, pages 165-174 (March, 1965); Tappi 48, No. 4, pages 214-230 (April, 1965); Tappi 48, No. 5, pages 270-272 (May, 1965) and Tappi 48, No. 8, pages 447-451 (August, 1965), in the impregnation stage the higher the impregnation temperature, the more complete and more uniform is the impregnation of the cooking chemicals into the wood chips, although at temperatures above 150° C. significant delignification occurs, which is undesirable. It is critical to the rapid vapor phase process of Kleinert to obtain in the impregnation stage the most complete and uniform impregnation practically possible for the chips and it is found that the more incomplete the impregnation the greater the screen rejects and thus the more limited the utility and applicability of the pulp in commercial processes. Kleinert in this regard teaches an optimum temperature in the range 130° to 140° C. for the impregnation stage.

In the impregnation of the dry chips with the kraft liquor Kleinert also teaches that the higher the concentration of the effective alkali in the impregnation liquor the better the impregnation, but conversely, the higher the concentration of effective alkali in the impregnation liquor the lower the strength of the pulp obtained, and with effective alkali concentrations in excess of 40 grams per liter the strength of the pulp is lowest. Kleinert still further teaches that the higher the liquor-to-wood ratio the better the impregnation obtained in the impregnation stage and in order to keep the concentration of alkali as low as possible Kleinert recommends a dynamic liquor-to-wood ratio as high as possible. However, even under such conditions, in order to provide a practically useful impregnation of the wood chips within a reasonably short period of time, of the order of 15 to 20 minutes, the impregnation of the chips at the aforesaid elevated temperature should be effected under superatmospheric pressure, optimally about 150 psig.

Kleinert thus proposes the following optimum conditions for the impregnation stage of his process to obtain a uniform and complete impregnation of the wood chips: an effective alkali concentration of the kraft liquor below 40 grams per liter, a dynamic liquor-to-wood ratio as high as possible, an impregnation temperature of 130° to 140° C., with the impregnation pressure above 150 psig, and an impregnation time of the order of 15 to 20 minutes.

In the subsequent vapor phase cooking stage, the impregnated chips, after removal of the excess of liquor, are very rapidly raised to a high temperature. The higher the temperature of cooking the lower the value to which the lignin content of the wood chips may be lowered within a short period of time by "bulk delignification", as opposed to "residual delignification", and as it is found that substantially small amounts of carbohydrate are consumed during the "bulk delignification", whereas during "residual delignification" comparatively large amounts of carbohydrate are removed per unit of lignin removed, the loss of carbohydrate in the vapor phase cooking stage is substantially lowered, with an improved yield over the conventional liquid phase process, in which the temperature is raised to the maximum high temperature over a relatively longer period of time. Kleinert proposes that the optimum conditions for the vapor phase cooking of the impregnated chips should be: a temperature of 182.5° C. with steam at a superatmospheric pressure of 140 psig and a cooking time of 20 minutes.

By this process Kleinert produces a cellulosic pulp with a lower lignin content and a higher carbohydrate-to-lignin ratio in a good yield, which pulp may be rapidly bleached to a high pulp brightness. In addition to the product advantages, the process, as set forth by Kleinert, has the advantages of being short, having a low chemical consumption and providing a black liquor of high concentration at a high temperature which allows for cheaper recovery of the chemicals from the black liquor.

However, it is found that while the process of Kleinert has the aforesaid process advantages the pulp obtained, while having a lower lignin content and a higher ratio of carbohydrate to lignin, has a number of properties which are inferior to conventional kraft pulp, which inferior properties have severely limited the commercial applicability of the process. In particular the pulp obtained in the Kleinert process has very limited uses as it has a high screen rejects content, a low C.E.D. viscosity, a reduced toughness and a reduced tensile and tear strength when compared with conventional kraft pulp prepared by liquid phase techniques known to those skilled in the art.

The present invention provides an improvement in the aforesaid vapor phase alkaline cooking process in which the screen rejects are substantially reduced and the further properties of the pulp are substantially comparable with conventional kraft pulp, and have essentially the same utility as conventional kraft pulp. At the same time the pulp yield obtained is substantially increased over and above that obtained in the Kleinert vapor phase process.

It has now been found according to the present invention that when in the aforesaid Kleinert process the impregnation of the wood chips is effected upon wood chips which have previously been saturated with water, then the impregnation of the chips may be effected within a reasonable period of time at atmospheric pressure and at a substantially lower temperature below 110° C., desirably in the range 60° to 90° C. and more desirably in the range 70° to 90° C.

Thus, applicants have found that when a prolonged impregnation time is applied to the chips at a temperature of 130° to 150° C. for the purpose of obtaining sufficient impregnation of the chemical, a large amount of nonlignin components, i.e. carbohydrates of the lignocellulosic material, are dissolved by the liquor during the impregnation stage and the delignification during this impregnation period of insignificant. A temperature of 130° to 150° C. is not favorable for pulping, yet at temperatures above about 110° C. the rate of removal of the carbohydrate from the wood chips becomes substantial and increases at a rate, up to a temperature in the range of about 150° to 160° C., greater than the rate of removal of the lignin. However, when dry chips are used, mere reduction of the impregnation temperature does not achieve, even under high pressure for extended periods of time, sufficient impregnation of the chips to avoid the completely unacceptable amount of screen rejects in the pulp obtained, which severely limits the utility of the pulp as aforesaid. Thus whereas it has been found by the applicants that it is practically impossible to impregnate a sufficient amount of chemicals into dry wood chips at a low temperature below 110° C. even under very high pressure within a short period of time such as 20 minutes, and that at a high alkali concentration intended to promote the uptake of chemicals into the wood there is a non-uniform distribution of the chemical between the outer and central part of the chips, which tends to deteriorate the outer parts of the chips, they have found that with water-saturated chips it is possible to impregnate the chips at atmospheric pressure within a practically acceptable period of time of the order of 60 minutes and obtain an essentially complete and uniform impregnation of the chips with uptake of a sufficient amount of chemicals by the chips for effecting the subsequent alkaline delignification. In particular, in the process of the present invention using water-saturated chips which provides for good impregnation of the chips there are negligible screen rejects and a higher carbohydrate-to-lignin ratio in the resultant pulp, giving a higher pulp yield and a corresponding lower alkali consumption in the pulping liquor and also a higher C.E.D. viscosity in the pulp obtained.

According to the present invention therefore there is provided in a process for the production of a chemical cellulosic pulp from a lignocellulosic material which comprises impregnating said lignocellulosic material in subdivided form, suitably in the form of chips, with an alkaline cooking liquor, in particular a kraft cooking liquor, removing the excess of cooking liquor from said impregnated material, contacting said material with steam at superatmospheric pressure to effect rapid heating thereof to cooking temperature, delignifying said impregnated material and passing the delignified material into an aqueous processing liquor, the improvement in which is that the subdivided material impregnated with said liquor has been previously saturated with water, whereby said impregnation is effected at a temperature below about 110° C.

As used herein the term "water-saturated material" refers to subdivided material, e.g. chips, in which at least the centers thereof are saturated with water and for softwoods the water content is usually in the range of 180 to 200 percent by weight (ovendry wood), while for hardwoods the water content is in excess of 150 percent by weight (ovendry wood). The moisture content of the chips can be raised to these levels, i.e. the chips can be saturated, by merely soaking the chips in warm water for a sufficient period of time. Generally the chips have a moisture content in excess of 170 percent by weight, based on the ovendry weight of the chips.

In the impregnation stage the chips are suitably impregnated with the alkaline cooking liquor at a temperature in the range of 70° to 90° C., preferably 80° to 90° C., for a period of from 0.5 to 2 hours, more usually for about an hour, at atmospheric pressure.

After draining the excess of liquor from the impregnated chips they are subjected to delignification by contact with steam at superatmospheric pressure, usually of the order of 115 to 150 psig, at a temperature suitably in the range 170° to 175° C. and whereas as aforesaid the optimum conditions taught by Kleinert are a temperature of about 182.5° C. and a period of 20 minutes, it has been found according to the present invention that by increasing the heating time such that the H factor, which for Kleinert's process is about 1,000, is increased in excess of 1,000 and suitably to at least 2,000, the pulp yield at constant Kappa number is even further increased, the C.E.D. viscosity of the pulp obtained is also increased and the alkali consumption is further decreased. Thus, whereas Kleinert is essentially concerned with obtaining a rapid vapor phase process and as such teaches an optimum temperature and time of 182.5° C. and 20 minutes, respectively, applicants have found that by increasing the time at which the pulp is maintained at the cooking temperature such that the H factor which is 1,000 for Kleinert is increased desirably up to and above 2,000, the properties of the pulp obtained and the yield of pulp obtained is also substantially enhanced. The H factor is an integrated value of cooking time and cooking rate at a given temperature and is more fully discussed in a technical report entitled "The H Factor: A Means of Expressing Cooking Times and Temperatures as a Single Variable" by K. E. Vroom, Pulp Paper Mag. Can., 58 (3): 228 (Convention Issue, 1957).

It has further been found according to the present invention that at the same H factor it is also possible to increase the C.E.D. viscosity of the pulp at the same Kappa number with substantially no loss in yield and substantially no increase in alkali consumption, by lowering the cooking temperature from 185° C. to 175° C. Further, although by increasing the H factor at the same temperature the brightness of the pulp obtained is slightly reduced, it is found that when the cooking temperature is lowered from 185° to 175° C. at the same H factor, the pulp brightness increases. In general therefore, it has been found by the applicants that by increasing the H factor above 1,000 and desirably to at least 2,000 and either at the same time or alternatively thereto reducing the temperature of cooking, the properties and the yield of the pulp obtained are substantially improved and at the same time, due to the increased retention time of the pulp in the vapor phase stage of the process from about 20 minutes to 40 or even 90 minutes when the temperature is lowered, it is possible to use with minor modification a continuous digester of the vertical type in common use in many existing kraft mills or alternatively a batch digester in an existing conventional liquid phase kraft mill for the process. Thus in the rapid vapor phase kraft process of Kleinert with a retention time of 20 minutes under pressure in the impregnation stage and 20 minutes in the vapor phase stage a conventional batch digester or a continuous digester of the vertical type in common use in existing mills is not suitable. With the process of the present invention, in which the impregnation stage does not require a pressure vessel, since it can be effected at atmospheric pressure at 90° C., the wood chips may be cooked in any ordinary digester, either continuous or batch, for 90 minutes at 175° C. and at 115 psig., followed desirably by diffusion washing or cold blow in the same digester such as a Kamyr digester.

Alternatively the process of the present invention can be effected in the Kleinert apparatus using the quenching technique proposed by him i.e. by passing the delignified material to a wash zone, introducing an aqueous processing liquid into said zone and passing said processing liquid in countercurrent contact with the delignified material to terminate the delignification of the material and to remove a portion of the solubilized amorphous substances of said material,

withdrawing the upper portion of the wash zone aqueous processing liquid having solubilized amorphous substances dissolved therein, and controlling the rate of withdrawal to provide an aqueous processing liquid having a concentration ranging from about 15 to about 30 percent of solubilized substances.

The present invention will be further illustrated by way of the following examples.

EXAMPLE 1

Red spruce guillotine chips with a size of 1 inch by 1 inch (grain direction) by one-eighth inch thick were prepared from red spruce logs. The chips were dried in the atmosphere to a moisture content of 10 percent based on dry wood, and were separated into aliquots of 280 gms. (ovendry weight). Each aliquot was saturated with water in a pressure vessel by two steam purges at 30 psig for 2 minutes each followed by impregnation of water at 70° C. under a pressure of nitrogen gas at 100 psig. for 1 hour. Following this the chips were sealed in a polyethylene bag and were stored in a cold room at 3° C. to 5° C. for from 2 to 5 weeks before being subjected to cooking. Before subjecting the chips to cooking, any free water was drained away by keeping the wet chips in a wire mesh basket (60 mesh) for 1 hour. While the surface of the chips was tending to dry the moisture content of the chips was 179 ± 3 percent, based on dry wood, which approached the saturation point for spruce wood of 190 to 200 percent.

In a series of vapor phase kraft cooks a number of these aliquots were subjected in an impregnation stage to heating with 1,120 ml of kraft liquor at a liquor-to-wood ratio of 4 to 1. The chips in cooks 238, 219, 216, 224, 225, 226, 227, 245, 228 and 229 were impregnated over a period of 60 minutes at a temperature of 90° C. at atmospheric pressure. In these cooks, after impregnation of the chips and removal of the excess of liquor, the impregnated chips were then subjected to steaming and in cooks 238, 219 and 206 the temperature of the impregnated chips was initially raised to 100° C. for 20 minutes and subsequently to 175° C. for 90 minutes, giving an H factor for the cooking process of 2,000. In cooks 224 and 225 the temperature of 175° C. was maintained for 40 minutes, giving an H factor for the cooking processes of 1,000. In cooks 226, and 227 the final temperature was 185° C. which was maintained for 40 minutes, giving an H factor for the cooking processes of 2,000. In cooks 245, 228 and 229 a cooking temperature of 185° C. was maintained for 20 minutes, giving an H factor of 1,000.

In cooks 241, 230 and 231 the moisture-saturated chips were heated over a period of 40 minutes to 140° C. during the impregnation with kraft liquor and were maintained at that temperature for 30 minutes. In cooks 232 and 233 the temperature was subsequently raised to 175° C. by steaming and was maintained at that temperature for 90 minutes, giving an H factor of 2,000 for the cooking process. In cooks 241, 230 and 231 the temperature was subsequently raised to 185° C. for 20 minutes by steaming, giving an H factor of 1,000 for the

cooking process. The conditions for the impregnation stage are set forth in the following Table 1 and the properties and yield of the pulps obtained are also set forth in Table 1.

	Consumed Eff. Alkali % on wood, as Na ₂ O	Screened Pulp Yield %	Re- jects %	Total Pulp Yield %	Kappa No.	Bright- ness
	13.2	50.0	1.1	51.1	41.7	25.6
10	14.4	48.6	0.4	49.0	30.7	29.2
	17.5	48.0	0.0	48.0	23.5	29.4
	17.6	48.3	0.3	48.6	30.4	32.3
	21.9	46.6	0.1	46.5	22.7	35.0
	14.5	48.7	0.0	48.7	28.0	28.2
	16.9	47.2	0.0	47.2	21.0	30.6
15	14.3	50.6	1.0	51.6	43.2	28.6
	17.8	47.8	0.0	47.8	25.7	33.2
	22.0	45.4	0.1	45.5	20.7	35.7
	14.5	48.8	1.3	50.1	43.6	30.4
	16.6	46.8	0.3	47.1	28.1	34.3
	19.2	45.0	0.1	45.1	21.5	37.1
20	12.2	49.3	3.6	52.9	68.6	24.6
	14.0	47.8	0.4	48.2	37.7	30.3
	17.2	45.9	0.1	46.0	27.8	33.6

In a comparative series of cooks, 280 gm. of chips with a comparatively low moisture content, to wit, 40 percent based on the ovendry weight of the chips, were initially subjected to two steam purges each of 3 minutes duration at 30 psig. and were impregnated with 1,400 cc of a kraft liquor at a liquor-to-wood ratio of 5 to 1. The chips were then separated into aliquots and these aliquots were subjected to cooks in which they were initially heated over 25 minutes to 140° C., maintained at that temperature for 30 minutes, subsequently heated to 185° C. by steam heating and were then maintained at that temperature for 20 minutes, giving an H factor of 1,000. The conditions of impregnation and the properties and yield of the pulp obtained are also given in Table 1. In each cook on termination of the steaming the delignified chips were quenched in water, disintegrated, and the pulp was screened through a vibrating flat screen with 0.010 inch slits and the weights of the screen rejects and the screened pulp were determined. These are summed up in Table 1 as the total pulp yield. From the above results graphs were plotted (the Figure) of total pulp yield, percent, against Kappa number at different H factors for the process according to the present invention (VKP) at various H factors, as well as for the process according to Kleinert (RVPK) using chips with an initial moisture content of 180 percent based on their ovendry weight, and impregnating at 140° C. in the impregnation stage, and also for the process according to Kleinert using comparatively dry chips, initial moisture content 40 percent based on ovendry weight.

It will be seen from the Figure that the process of the present invention is superior to the conventional kraft process, which is operated in the liquid phase, and is also superior to the Kleinert process and even to the Kleinert process using moisture-saturated chips. To illustrate more clearly the advantages of the present invention from the Figure and Table 1,

TABLE 1

Cook number	Vapour phase kraft	Chip mois- ture based on dry wood, percent	Impreg- nation temper- ature, ° C.	Vapour phase cooking tempera- ture, ° C.	"H" factor in vapour phase stage	Liquor- to- wood ratio	Eff. alkali conc., g./l. as Na ₂ O	Sulph- idity, per- cent	NaOH conc., g./l. as Na ₂ O	Na ₂ S conc., g./l. as Na ₂ O
1.....	238	180	90	175	2,000	4/1	68	30	56.0	24.0
2.....	219	180	90	175	2,000	4/1	75	30	61.9	26.5
3.....	206	180	90	175	2,000	4/1	90	30	74.1	31.8
1.....	224	180	90	175	1,000	4/1	95	30	78.3	33.5
2.....	225	180	90	175	1,000	4/1	120	30	98.8	42.4
1.....	226	180	90	185	2,000	4/1	75	30	61.9	26.5
2.....	227	180	90	185	2,000	4/1	90	30	74.1	31.8
1.....	245	180	90	185	1,000	4/1	75	30	61.8	26.5
2.....	228	180	90	185	1,000	4/1	95	30	78.3	33.5
3.....	229	180	90	185	1,000	4/1	120	30	98.8	42.4
1.....	241	180	140	185	1,000	4/1	65	30	53.5	23.0
2.....	230	180	140	185	1,000	4/1	80	30	65.9	28.2
3.....	231	180	140	185	1,000	4/1	95	30	78.3	33.5
1.....	14	40	140	185	1,000	5/1	38	30	31.1	13.3
2.....	15	40	140	185	1,000	5/1	49	30	40.4	17.4
3.....	16	40	140	185	1,000	5/1	63	30	52.0	22.2

Table 2 was prepared, showing the yields and 0.5 percent C.E.D. viscosities of certain pulps at Kappa No. 30. It will be readily seen from this Table that the lower the impregnation temperature, the lower the vapor phase cooking temperature and the higher the H factor, the better are the properties of the pulp obtained, in particular the pulp viscosity and pulp yield are higher, and the alkali consumption required to reach Kappa No. 30 is lower.

TABLE 2
Properties of pulps at kappa No. 30

Impregnation temperature, ° C.	Vapour phase cooking temperature, ° C.	"H" factor	Wood moisture, percent on dry wood	Total pulp yield, percent on wood	0.5% C.E.D. pulp viscosity, cps.	Alkali requirement, as Na ₂ O, percent on wood
140.....	185	1,000	40	46.5	14	16.4
140.....	185	1,000	180	47.4	16	16.5
90.....	185	1,000	180	48.5	15	16.5
90.....	175	1,000	180	48.4	17	17.6
90.....	185	2,000	180	49.1	19	14.0
90.....	175	2,000	180	49.1	22	14.6

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. In a process for the production of a chemical cellulosic pulp from a lignocellulosic material which comprises impregnating said lignocellulosic material in chip form with an alkaline cooking liquor, removing the excess of cooking liquor from said impregnated material, delignifying said impregnated material by rapidly heating said impregnated material with

steam and passing said delignified material into an aqueous processing liquor, the improvement in which the lignocellulosic material impregnated with said liquor is a water-saturated material and said impregnation is effected at a temperature below about 110° C.

2. A process as claimed in claim 1 in which the impregnation is effected at a temperature in the range 60° to 90° C.

3. A process as claimed in claim 1 in which the impregna-

tion is effected at a temperature in the range 70° to 90° C.

4. A process as claimed in claim 1 in which the delignification is effected at a temperature in the range 170° to 185° C.

5. A process as claimed in claim 1 in which the delignification is effected at a temperature of 170° to 175° C.

6. A process as claimed in claim 1 in which the impregnation liquor is a kraft liquor.

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