

Nov. 29, 1960

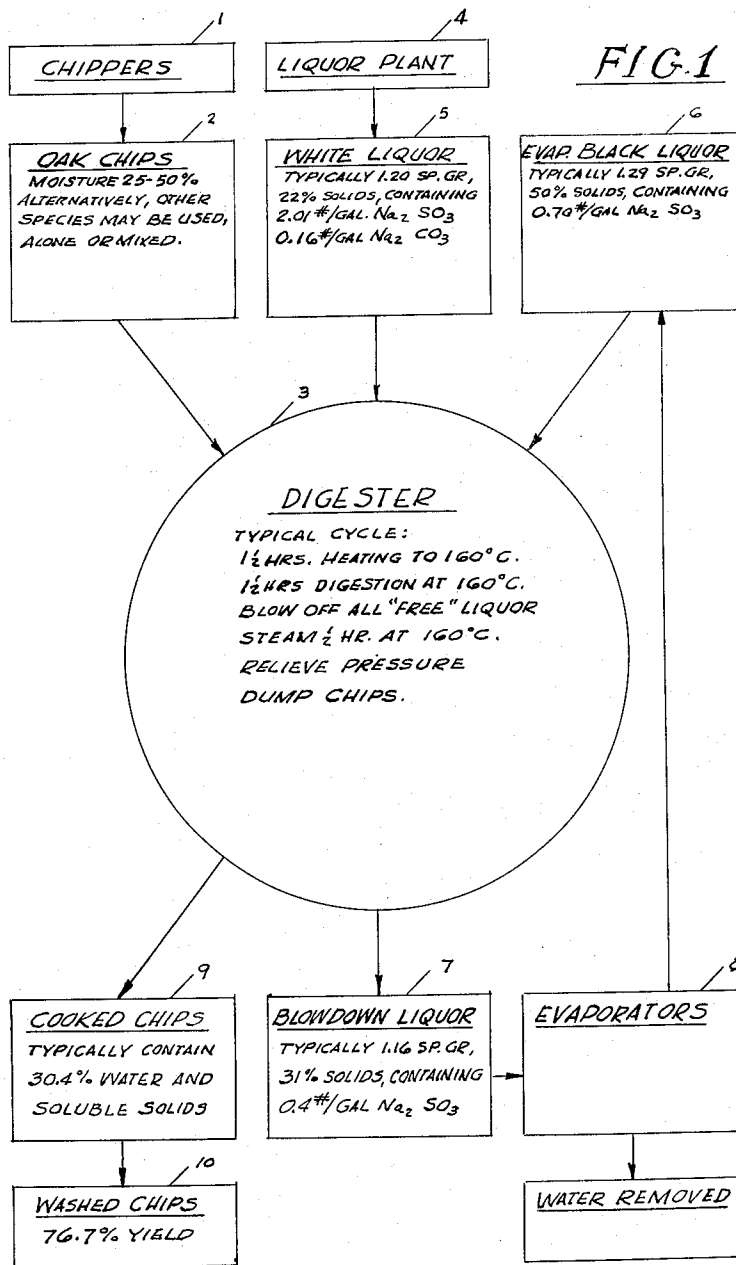
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2,962,412

PROCESS OF MANUFACTURING WOOD PULP

Filed Oct. 22, 1957

3 Sheets-Sheet 1



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3 Sheets-Sheet 2

FIG. 2

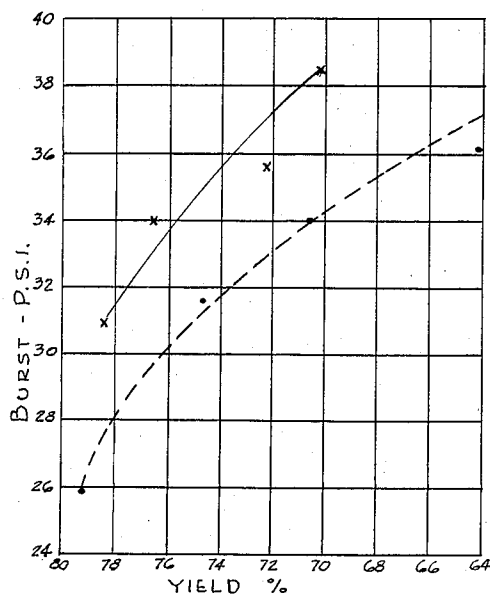
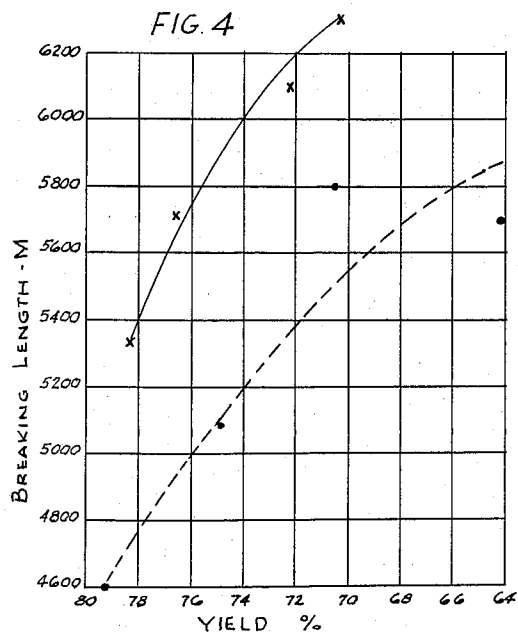


FIG. 4



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3 Sheets-Sheet 3

FIG. 3

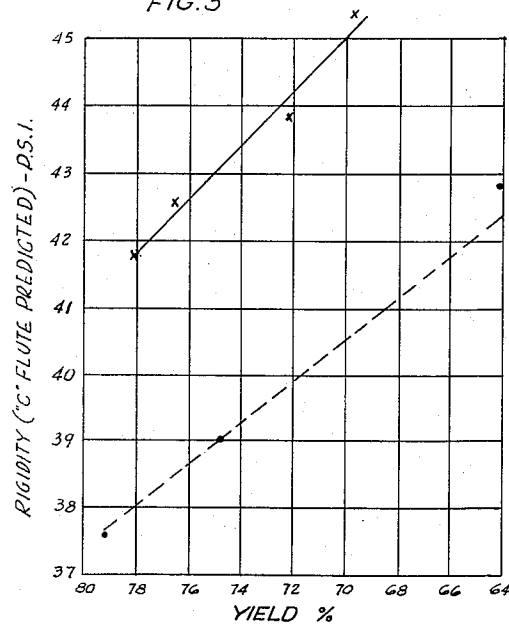
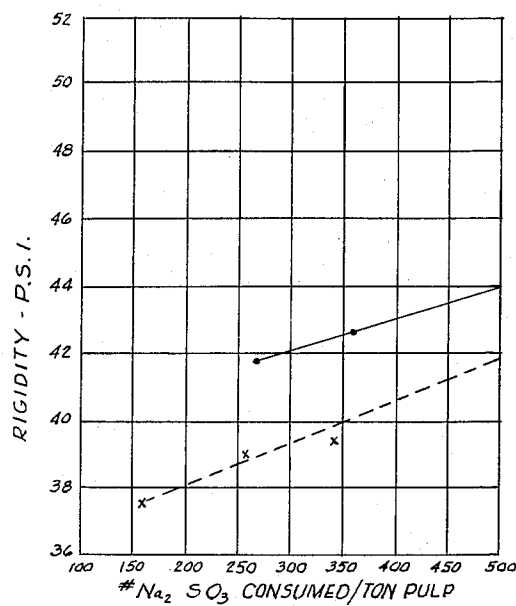


FIG. 5



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PROCESS OF MANUFACTURING WOOD PULP

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9 Claims. (Cl. 162—61)

This invention relates to the manufacture of wood pulp, and more particularly to the production of fibrous pulp using neutral digestion liquors.

One of the objects of this invention is to provide a pulping procedure utilizing neutral digestion liquors to effectively produce high quality pulp having good strength properties at significantly high yields.

Another object of this invention is to provide such a pulping procedure which is particularly effective with dense wood species.

A further object is to provide such a pulping procedure which includes a recovery system whereby the cooking chemicals may be recovered in such form that they are available and may be continuously reused in the pulping process of this invention.

Still another object is to produce pulp of high quality in high yield under conditions which permit comparatively small consumption of cooking chemicals and processing time.

Further objects and objects relating to process details and advantages of use of this invention will more definitely appear from the description to follow.

Numerous processes have been proposed for treating cellulosic materials to produce cellulose fibers from wood, whereby neutral or slightly alkaline alkali-metal monosulfite solutions have been utilized as the chemical pulping agent for the digestion of wood. In general, most of these processes do not provide for the absorption of an adequate amount of the chemical pulping agent, particularly by dense wood species. Conversely, while certain of these processes provide for increasing the amount of chemicals in attempting to solve the long recognized problem of providing separation of wood fibers by dissolving the intercellular material of the wood without causing substantial decomposition of the ligno-cellulose, and concomitantly producing a high yield of a good quality pulp, there has hitherto been no recognized solution of this problem as evidenced by the prevalence of unsatisfactory reduced yields of pulp. In addition, such resulting pulp exhibited inferior strength properties and in consequence is not considered entirely suitable for the manufacture of paperboard for corrugating and assembly into shipping containers.

This invention, in a simple preferred embodiment, may be practiced economically and satisfactorily using well known equipment and materials and with a controlled sequence in operation. The practicing of it is in a manner readily adaptable to pulp mills equipped for the so-called "soda-process," Figure 1 of the drawing being a diagrammatic representation of a flow sheet showing the practicing of the present invention.

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The present invention embodies a process of manufacturing wood pulp, and contemplates the use of a substantial excess of cooking chemicals, at comparatively high concentration, whereby the pulping reaction is accelerated and wood-chip cooking is expeditiously effected at a comparatively low temperature, resulting in high pulp yield of quality pulp. Accordingly, wood chips are charged into a suitable digester along with a solution comprising an alkali-metal monosulfite. The digester and its contents are brought rapidly to a suitable cooking temperature and then held at this temperature for a period of time, following which all of the liquor in the digester, except that adsorbed in the wood chips, is removed and the digestion continued in the vapor phase for an additional time interval, following which the pressure on the digester is relieved and the cooked chips are removed.

The spent liquor removed from the digester is concentrated as by evaporation and re-used in subsequent digestions, being admixed with additional amounts of fresh alkali-metal monosulfite solution. The cooked chips are defibered in a suitable attrition device and washed free of soluble solids, yielding a pulp in unusually high yield and of excellent quality.

Referring to Figure 1, wood is reduced to chips in a conventional manner as by suitable chippers 1. The species of wood is not of particular concern, the process being particularly effective with dense hardwoods, such as oak, but is also equally effective with other species, and is well adapted to the pulping of mixtures of wood species. Likewise, coniferous woods such as pine may be treated effectively. The aforesaid wood chips, which are suitably of conventional size for pulping purposes (i.e. 1/4" to 1" in length) and normally contain 25 percent to 50 percent moisture as indicated at 2 are charged to suitable digester 3. The digester 3 is a pressure vessel of suitable construction and may be lined with stainless steel, if desired, although the digestion liquors of this process are but slightly corrosive, and such linings may be eliminated in the interests of reduced cost.

White liquor is prepared in a suitable liquor plant 4 and has a typical composition, as indicated at 5, of 22 percent solids containing 2.01 lb. sodium monosulfite and 0.16 lb. of sodium carbonate per gallon. Specific gravity of the white liquor is typically 1.20. In place of sodium monosulfite, other alkali metal monosulfites, such as potassium monosulfite may be used. The said white liquor together with a suitable quantity of evaporated black liquor 6 is added to the digester 3 so as to provide a final mixture containing 125 gm. per liter of sodium monosulfite. A total amount of sodium monosulfite amounting to 30 to 50 percent of the weight of oven dry wood chips makes up the digester charge. The evaporated black liquor 6 typically has a specific gravity of 1.29, and a solids content of 50 percent, containing 0.70 lb. per gallon of sodium monosulfite.

This feature of recycling the spent or black liquor is optional, and serves the purpose of reducing cost by reusing the sodium monosulfite it contains, concurrent with reduction in the potential stream pollutional load which would result, were it to be discarded.

In carrying out the pulping procedure of this invention, the digester 3, containing the woody material in the form of wood chips and the digestion liquor, is then suitably closed and its contents are subjected to rapid cooking

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therein, as by steaming, to the desired digestion temperature, which is selected in accordance with conditions used for other variables, but generally lies between 140 and 170° C., suitably 160° C., it being understood that, for equivalent yield and quality of pulp, a lower temperature requires a longer time of digestion while a higher temperature requires a shorter time. The time required to heat the digester 3 and its contents to digestion temperature will vary somewhat, depending on its size, but a typical time for such heating is 1.5 hours. Once this temperature is attained, it is maintained for an additional time interval, for example, 1.5 hours, and then the free liquor is removed, leaving only that liquor which is retained by the chips. Following removal of the free or blow-down liquor, the digester and contents are heated, as by steaming, for an additional time interval, suitably 0.5 hour, after which the pressure is relieved and the softened chips are removed.

The blow-down liquor, after removal from the digester has typically a specific gravity of 1.16. This liquor contains 31 percent solids and 0.4 lb./gal. of unreacted sodium monosulfite, as indicated at 7. Such blow-down liquor is concentrated by evaporators 8 and may then be used with white liquor for a subsequent digester or may be delivered to a chemical recovery operation.

The cooked chips, which typically contain 30.4 percent water and soluble solids, as shown at 9, are defibered in a suitable attrition mill, and then washed, giving washed pulp 10 in a yield of 76.7 percent (oven-dry basis).

By adjusting the digestion conditions in the practice of this invention, as by reducing digestion time, it has been found that the yield of pulp may be increased to as high as 80 percent or more, with no substantially unsatisfactory degradation in pulp quality. Moreover, higher temperatures during digestion, or longer time of digestion have resulted in reduced yields, but with an improvement in pulp quality, particularly with regard to crush resistance when the pulp is subsequently converted to a corrugated medium and assembled in a shipping container. However, temperatures much in excess of 160° C. appear to have a marked deleterious effect on pulp quality as determined by rigidity burst or tensile strength, and such temperatures are to be avoided. Generally speaking, digestion conditions are selected to give the maximum yield of pulp consistent with the desired pulp quality levels.

The following examples, practiced during commercial operations, are presented in illustration but not in limitation of the present invention. In such operations, a considerable quantity of oak chips were prepared and, after screening to remove oversize and fine fragments, the chips were blended and stored for use as hereafter set forth.

EXAMPLE 1

A charge of these chips was placed in a digester and a cooking liquor, utilizing blow-down liquor from a previous cook and containing 125 grams per liter of Na_2SO_3 and 7 grams per liter of Na_2CO_3 , was added in sufficient amount to provide a chemical ratio of 42.5 percent (based on O.D. wood) and a liquor ratio of 3.8 parts liquor to 1 part O.D. wood. This charge was heated to 160° C. in 0.5 hour and maintained at 160° C. for an additional 0.5 hour. At this point, all the liquor was removed from the digester, except that adsorbed in the chips, and the charge steamed for an additional 0.5 hour at a temperature of about 160° C., and a pressure of approximately 105 p.s.i., following which the pressure was relieved and the cooked chips removed. After defibering in a disc mill and washing, pulp in a yield of 78.2 percent (based on O.D. wood) was obtained.

Analysis of the spent liquor indicated that 10.7 per-

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cent Na_2SO_3 (based on O.D. wood) had been consumed during the cook. A portion of this pulp was also analyzed chemically for its content of lignin, pentosans and alpha cellulose and the results thereof are shown in Table A, *infra*. Moreover, a portion of this pulp was made into sheets of paper, tested as to bursting strength, tensile and rigidity and the results thereof are also shown in Table A.

EXAMPLE 2

A charge of oak chips as prepared for Example 1 was treated under the conditions of Example 1, except that heating time to 160° C. amounted to 1.5 hours, and the charge was held at 160° C. for 1.5 hours before removing the blow-down liquor. Steaming after removal of the liquor continued for 0.5 hour. The yield of pulp was 76.7 percent and the Na_2SO_3 consumed amounted to 13.5 percent (based on O.D. wood). Quality of this pulp was also fully up to the requirements of a commercially acceptable corrugating board as shown by Table A, *infra*.

EXAMPLE 3

A charge of oak chips as prepared for Example 1 was treated under the conditions of Example 2, except that, after heating the charge to 160° C. in 1.5 hours, the charge was held at this temperature for 3 hours before removing the blow-down liquor. Steaming in the vapor phase continued for an additional 0.5 hour after removal of the liquor. The yield of pulp was 72.1 percent and the Na_2SO_3 consumed amounted to 19.4 percent (based on O.D. wood). Strength of this pulp was superior to that of Examples 1 and 2, as shown by Table A.

EXAMPLE 4

A charge of oak chips as prepared for Example 1 was treated under the conditions of Example 2, except that, after heating the charge to 160° C. in 1.5 hours, the charge was held at this temperature for 4.5 hours before removing the blow-down liquor and steaming the vapor phase for an additional 0.5 hour. The yield of pulp was 70.1 percent and Na_2SO_3 consumed amounted to 19.0 percent (based on O.D. wood). The quality of this pulp showed further improvement over that of Example 3.

Illustratively, the following table sets forth the conditions and properties which have actually been obtained by this invention in the production of high pulp yield, quality pulp as established by the foregoing examples:

Table A

Example.....	1	2	3	4
Na_2SO_3 Consumed, percent.....	10.7	13.5	19.4	19.0
Yield, O.D. pulp, percent.....	78.2	76.7	72.1	70.1
Lignin, percent.....	11.3	10.1	7.4	9.3
Alpha Cellulose, percent.....	55.4	52.3	49.8	46.2
Pentosans, percent.....	16.1	15.8	14.6	14.5
Bursting Strength, p.s.i.....	31.1	34.0	35.5	38.5
Tensile, breaking length, meters.....	5,325	5,700	6,100	6,300
Rigidity ("C" flute predicted), p.s.i.....	41.9	42.7	43.8	45.4

In the above computation, Standard Methods of the Technical Association of the Pulp and Paper Industry (TAPPI) have been employed for the determination of lignin, alpha cellulose and pentosans. These values, as well as those given for Na_2SO_3 consumed and for yield are based on the O.D. weight of wood in the digester charge.

Physical strength data was obtained from handsheets, prepared by the TAPPI standard methods, except that basis weight was increased to 26 lb. per 1000 sq. ft. Burst and tensile strength were determined by TAPPI procedures, while rigidity was determined by the Con-

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córa Medium Fluter, and converted to units of "C" flute predicted crush resistance in pounds per square inch.

Likewise, for the purposes of comparison as between the present process of providing a pulp by pulping procedure utilizing neutral digestion liquors as shown by Table A, and pulp produced in accordance with former practices using a comparatively limited amount of cooking chemicals at relatively low concentrations as used in typical prior commercial operations, the following figures, as tested by previously mentioned TAPPI procedures, are given by way of illustration to show comparative physical strength data:

Table B

	Old Commercial Sample			
	a	b	c	d
Bursting Strength, p.s.i.	26.0	31.6	34.0	36.2
Tensile, breaking length, meters	4,600	5,100	5,800	5,700
Rigidity ("C" flute predicted), p.s.i.	37.5	39.0	39.2	42.8
Na ₂ SO ₃ Consumed, percent	6.0	9.4	12.3	18.8
Yield, O.D. Pulp, percent	79.3	74.8	70.5	64.1

In Figures 2 to 4, inclusive, of the drawings, the strength comparisons of Tables A and B, have been graphically shown, the solid line presentations being in accordance with this invention, whereas, the broken line presentations are in accord with the data computed by testing papers produced from pulp obtained by old commercial neutral digestion liquor processes.

In Figure 5 of the drawings, the rigidity comparisons of Tables A and B, have been graphically shown in relation to the amount of sodium sulfite consumed per ton of dry pulp. Again the solid line presentation is in accordance with this invention, whereas the broken line presentation is in accord with the data computed by testing pulp and papers produced from pulp obtained by old commercial neutral digestion liquor processes. Thus, it will be observed that for a given level of rigidity, the process of the present invention consumes less sodium sulfite than the old commercial methods, which constitutes a distinct economic advantage.

While the foregoing presents preferred embodiments of the present invention, it is obvious that other conditions and/or equivalents may be employed without departing from the scope of the invention, which is defined in the appended claims.

What is claimed is:

1. A process for the production of wood pulp which comprises treating wood chips in a digester with a neutral liquor containing essentially sodium sulfite in an amount to provide a chemical ratio of the order of 42.5 percent sodium sulfite and a liquor to wood ratio of the order of 3.8 parts liquor to 1 part of wood, cooking said wood chips with said liquor under temperature conditions of the order of 160° C. for at least 0.5 hour and not more than 4.5 hours, removing the liquor and thereafter continuing the cooking of the wood chips in the vapor phase for about 0.5 hour whereby a high yield of relatively high strength pulp is produced with a minimum consumption of said sodium sulfite.

2. A process for the production of wood pulp which comprises treating wood chips of dense wood species in a digester with a neutral liquor containing essentially sodium sulfite in an amount to provide a chemical ratio of the order of 42.5 percent sodium sulfite and a liquor to wood ratio of the order of 3.8 parts liquor to 1 part of wood, cooking said wood chips with said liquor under temperature conditions of the order of 160° C. for at least 0.5 hour and not more than 4.5 hours, removing the liquor and thereafter heating the wood chips to a temperature of about 160° C. for about 0.5 hour whereby a

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high yield of relatively high strength pulp is produced with a minimum consumption of said sodium sulfite.

3. A method of digesting fibrous materials which comprises introducing the fibrous material and a neutral digestion liquor into a digester to provide a liquor to material ratio of about 3.8 parts liquor to 1 part of material, heating said material and said liquor to a temperature of approximately 160° C. for 1.5 hours, maintaining said heating for an additional 1.5 hours to continue pulping reaction, removing the unadsorbed cooking liquor, and thereafter cooking the material in the vapor phase for about 0.5 hour whereby a high yield of relatively high strength pulp is produced with a minimum consumption of said liquor.

4. A process for the production of wood pulp which comprises treating wood chips of dense wood species with a neutral liquor containing essentially sodium sulfite in an amount to provide a chemical ratio of the order of 42.5 percent sodium sulfite, cooking said wood chips with said liquor under temperature conditions of the order of 160° C. for at least 0.5 hour and not more than 4.5 hours, removing the liquor and thereafter cooking the wood chips in the vapor phase for about 0.5 hour whereby a high yield of relatively high strength pulp is produced with a minimum consumption of said sodium sulfite.

5. A process for the production of wood pulp which comprises treating wood chips in a digester with a liquor containing essentially sodium monosulfite in an amount to provide a chemical ratio of the order of 42.5 percent sodium monosulfite and a liquor to wood ratio of the order of 3.8 parts liquor to 1 part of wood, cooking said wood chips with said liquor under temperature conditions of the order of 160° C. for at least 0.5 hour, removing the liquor and thereafter steaming the wood chips in the vapor phase for about 0.5 hour whereby a high yield of relatively high strength pulp is produced with a minimum consumption of said sodium monosulfite.

6. A process for the production of wood pulp which comprises treating wood chips in a digester with a liquor containing essentially sodium monosulfite in an amount to provide a chemical ratio of the order of 42.5 percent sodium monosulfite and a liquor to wood ratio of the order of 3.8 parts liquor to 1 part of wood, heating said wood chips with said liquor until not more than about 15 percent of sodium monosulfite is consumed, removing the liquor and thereafter steaming the wood chips in the vapor phase for about 0.5 hour whereby a high yield of relatively high strength pulp is produced with a minimum consumption of said sodium monosulfite.

7. A process for the production of wood pulp which comprises treating wood chips in a digester with a liquor containing essentially sodium monosulfite in an amount to provide a chemical ratio of the order of 42.5 percent sodium monosulfite and a liquor to wood ratio of the order of 3.8 parts liquor to 1 part of wood, cooking said wood chips with said liquor under temperature conditions of the order of about 140° to approximately 170° C. for at least 0.5 hour and not more than 4.5 hours, removing the liquor and thereafter cooking the wood chips in the vapor phase for about 0.5 hour whereby a high yield of relatively high strength pulp is produced with a minimum consumption of said sodium monosulfite.

8. A process for the production of wood pulp comprising treating wood chips of dense wood species in a digester with a neutral liquor containing essentially sodium monosulfite in a concentration of 125 grams/liter, cooking said wood chips with said liquor without effecting a separate preliminary impregnation under temperature conditions of from 140° C. to 170° C. for at least 0.5 hour and not more than 4.5 hours, removing the unadsorbed liquor and thereafter continuing the cooking of the said wood chips in the adsorbed liquor for about 0.5 hour whereby a high yield of relatively high strength

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pulp is produced with a minimum consumption of said sodium monosulfite.

9. A process for the production of wood pulp which comprises treating wood chips of dense wood species in a digester with a neutral liquor containing essentially sodium monosulfite in an amount to provide a chemical ratio of from 30 to 50 percent of sodium monosulfite based on dry weight of the wood chips, cooking said wood chips with said liquor under temperature conditions of the order of 160° C. for at least 0.5 hour and not more than 4.5 hours, removing the unadsorbed liquor and thereafter continuing the cooking of the wood chips in the

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adsorbed liquor for about 0.5 hour whereby a high yield of relatively high strength pulp is produced with a minimum consumption of said sodium monosulfite.

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UNITED STATES PATENT OFFICE
CERTIFICATION OF CORRECTION

Patent No. 2,962,412

November 29, 1960

Cloyd B. Richardson

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

In the grant, lines 2 and 12, and in the heading to the printed specification, lines 3 and 4, name of assignee, for "The Meade Corporation", each occurrence, read -- The Mead Corporation --.

Signed and sealed this 4th day of July 1961.

(SEAL)

Attest:

ERNEST W. SWIDER

Attesting Officer

DAVID L. LADD

Commissioner of Patents