

[54] PRESS ALKALINE EXTRACTION OF CELLULOSIC PULP

[75] Inventor: Norman Liebergott, Laval, Quebec, Canada

[73] Assignee: Pulp and Paper Research Institute of Canada, Quebec, Canada

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[51] Int. Cl. D21c 9/14

[58] Field of Search 162/89, 88, 67, 66, 90

[56] References Cited

UNITED STATES PATENTS

2,882,965	4/1959	Wayman et al.	162/89
3,575,795	4/1971	Brinkley et al.	162/88 X
3,586,599	6/1971	Yorston et al.	162/88 X
3,632,469	1/1972	Wilder	162/89 X

Primary Examiner—S. Leon Bashore
Assistant Examiner—Arthur L. Corbin
Attorney, Agent, or Firm—Millen, Rapses & White

[57] ABSTRACT

A procedure is provided for the alkaline extraction of cellulosic matter in the manufacture of paper-making grades of pulp which has been pretreated with a delignification agent and/or with an oxidizing reagent. The cellulosic matter may be bisulfite pulp or kraft chemical pulp. It has unexpectedly been found that when such a pretreated pulp, e.g. a chlorinated and washed pulp, is mixed rapidly with an aqueous solution of an alkali, e.g. sodium hydroxide, at a particularly defined concentration correlated to the Kappa number of the pulp and being within the range of about 0.5–5% by weight, the mixing being conducted at a consistency of up to about 12%, preferably at about 4% to about 12%, at a temperature of from about 40° to about 90°C., preferably at a temperature of from about 60°C to about 80°C., and is substantially immediately thereafter pressed to a pulp consistency of from about 18% to about 40%, preferably to a consistency of from about 20 to about 40%, then a single extraction treatment is as effective as conventional alkaline extraction (at 60° to 80°C. for 1 hour), in reducing the Kappa No. of a chlorinated pulp to similar levels. The procedure also is effective in the neutralization step between two or more oxidation stages. Little or no washing with water is necessary following an alkaline extraction by the process according to this invention.

20 Claims, 4 Drawing Figures

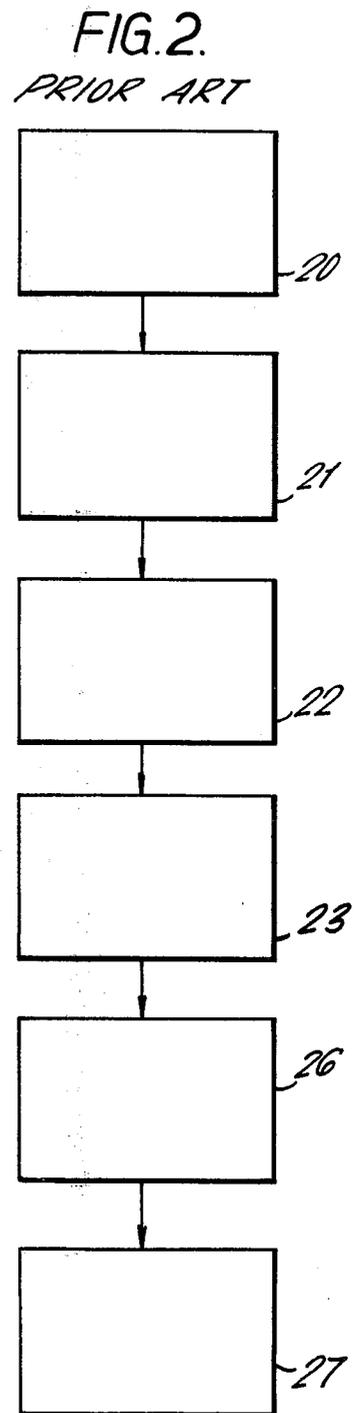
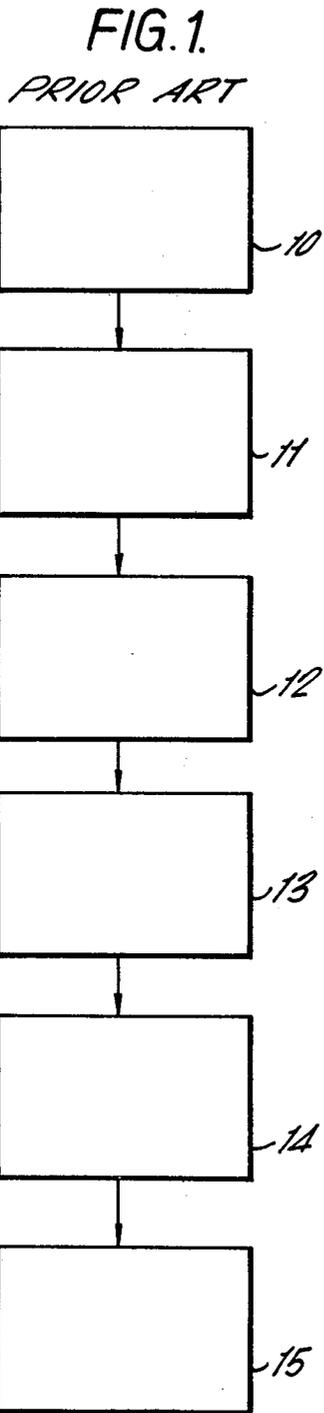


FIG. 3.
PRIOR ART

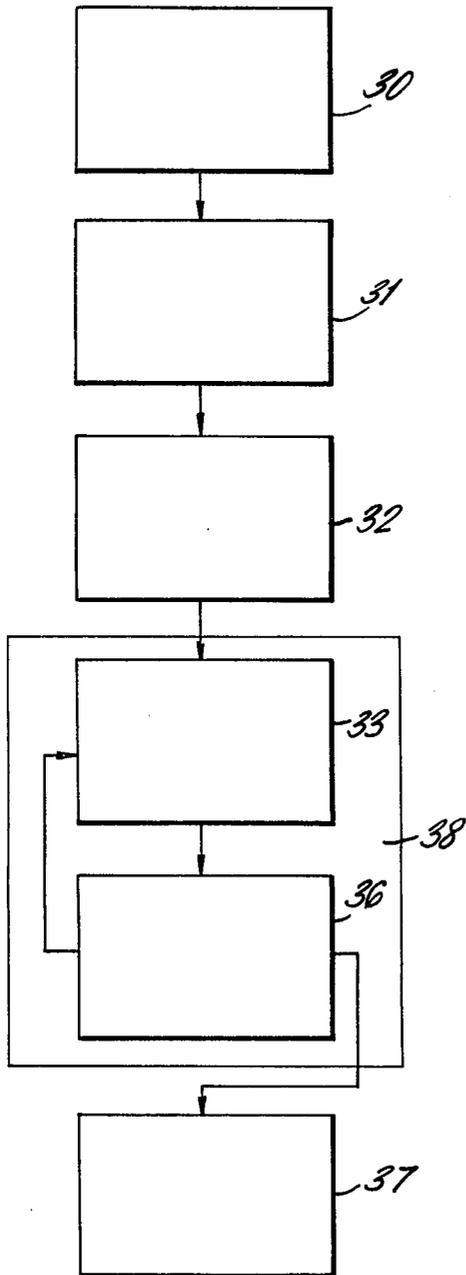
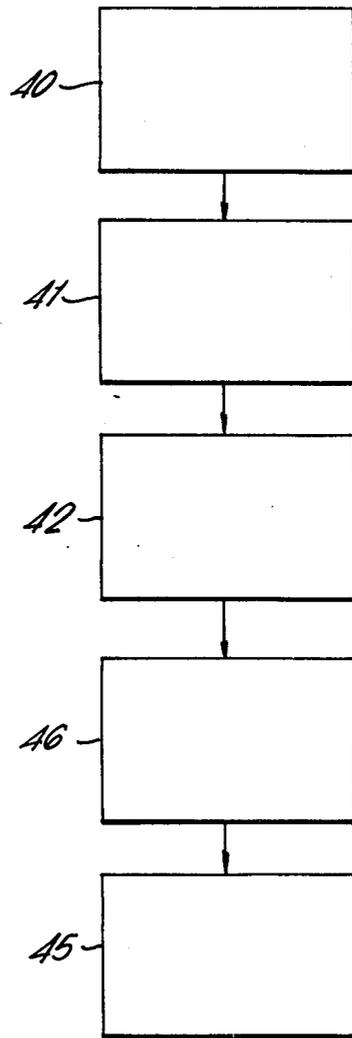


FIG. 4.



PRESS ALKALINE EXTRACTION OF CELLULOSIC PULP

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to improvements in the alkaline extraction of a wood pulp, in the manufacture of paper-making grades of pulp, after treatment with various bleaching chemicals such as, for example, elemental chlorine, chlorine monoxide, chlorine dioxide, hypochlorites, chlorites, peroxides, chlorates, bichromates, oxides of nitrogen and permanganates.

2. Description of the Prior Art

Bleaching is a continuation of the cooking process in which the ligneous material and colouring matter which remain in the chemical pulp are removed selectively with as little degradation of the pulp fibre as possible. Bleaching of the pulp has advanced to a high degree of sophistication involving single-stage and multi-stage procedures. Bleaching of a chemical pulp is accomplished in several stages. As known in the art, a "stage" constitutes a phase starting with the addition and reaction of a chemical with a pulp and ends with the washing of the pulp. The selection of a proper number of stages, and hence the desired sequence, is determined by (a) the nature of the fibre to be bleached (softwood or hardwood), (b) the pulping process which was applied (acid, neutral or alkaline), and (c) whether the pulp is to be used for the manufacture of paper-making grades of pulp, or the manufacture of dissolving pulps.

The first step usually consists of treating pulp in aqueous suspension with chlorine solution in the aqueous phase; an aqueous solution of chlorine dioxide may be used either in admixture with the chlorine or in replacement of the chlorine in this first stage. It is also known that, by use of a high consistency, gas phase bleaching technique, pulp at a high consistency (about 15 to about 60%) and in comminuted form, can be reacted with gaseous chlorine, chlorine monoxide, chlorine dioxide, ozone-peroxides and oxides of nitrogen. For the attainment of brightness, with strength preservation, for brightness stability and bleaching economy, the reaction products resulting from chlorination or delignification by oxidative bleaching, are removed as they are formed in those operations by means of an alkaline extraction. Caustic soda is a preferred agent, but other alkalis (e.g. ammonia, sodium carbonate, calcium hydroxide, etc.) have been suggested and used. This alkaline treatment on pulp is generally carried out in slurry form at low consistencies ranging from about 8 to about 15% and at times varying from about 60 minutes to about 2 hours, and at temperatures ranging from about 40°C to about 70°C.

In the manufacture of paper-making grades of pulp, for the attainment of brightness, with strength preservation, for brightness stability and bleaching economy, the reaction products resulting from chlorination and oxidative bleaching are removed as they are formed in such operation. The products are removed by treatment with strong bases in a separate treatment known as an "alkaline extraction". Such alkaline extraction generally follows the oxidative step. FIG. 1, to be described later, is a schematic flow diagram of a convention such alkaline extraction.

Many suggestions have been made for a rapid bleaching stage in the manufacture of paper-making grades of

pulp but most have relied on a rapid bleaching portion of the stage, with little or no regard to the post-bleaching alkaline extraction portions of the stage. For example, W. H. Rapson teaches, in "Dynamic Bleaching", TAPPI, Vol. 49, No. 8, pp. 324-344, 1966, that the limiting factor in the bleaching process is not the chemical reaction rate but, instead, is the rate of diffusion of the reactant to the reaction sites. Rapson proposed an improvement which involved passing the bleaching solution through a layer of pulp, thus displacing the liquid already present in the pulp layer. In this way, the bleaching solution would be in constant motion with respect to the pulp fibre. The rate of mass transfer through the water layer to the fibre wall would be greatly increased, and thus the bleaching time could be reduced from hours to minutes for each stage. However desirable that end might be, Rapson's proposal suffered from the deficiency that, in order to accomplish rapid bleaching by this dynamic technique, larger amounts of chemicals were required.

A. W. Brinkley, Jr. et al, in U.S. Pat. No. 3,575,795 issued Apr. 20, 1971, taught a utilization of Rapson's technique, increasing the consistency to between about 5% and about 15%. They thus provided a scheme for the perpetuation of the dynamic bleaching technique. However, such scheme still suffered the deficiency of large chemical requirements.

Allan G. Jamieson taught in Canadian Pat. No. 881,406 issued Sept. 21, 1971, a continuous process for rapid bleaching of a wood pulp by treating wood pulp in an aqueous suspension at a consistency of about 3% to about 6% by weight with reagents in a bleaching sequence which included the three portions of a stage of (1) treatment with chlorine, (2) alkaline extraction, and (3) treatment with chlorine dioxide, under conditions of continuous agitation in continuously stirred vessels, the residence time of the pulp in each portion of the stage not exceeding about 10 minutes, and separating the reaction products and unreacted reagent from the pulp between the portions of the stages. This patent thus suffered the deficiency of a relatively lengthy alkaline extraction period.

Finally, N. Liebergott and F. H. Yorston taught in "Experiments in Rapid High Density Bleaching", TAPPI Vol. 48, No. 1, pp. 20-24, 1965, press washing between portions of the stages of bleaching. After high density chlorination and after treatment with alkali, pulps were thickened in a press from about 10% consistency to about 30% consistency. In such publication, it is reported that some 70% of the soluble material in the stock is removed in this step. In order to complete the washing according to these teachings, the press cakes were again slurried briefly to about 10% consistency in water and thickened again. Dilution and pressing were repeated once more. Thus, in order to achieve at least 95% efficiency with respect to removal of electrolytes from the pulp, it was taught that three press washings were needed to replace conventional washing portions of the stage.

For dissolving purposes a pulp must be of high purity, (e.g. substantially free from ash, resin and hemicelluloses) and have the right reactivity and viscosity, and an alpha cellulose content in excess of about 91%. These objectives can be obtained by intensifying the alkaline extraction, in two modifications, known as hot caustic purification and cold caustic purification. FIG. 2, to be described later, is a schematic flow diagram of

a hot caustic purification, and FIG. 3, to be described later, is a schematic flow diagram of a cold caustic purification.

SUMMARY OF THE INVENTION

Aims of the Invention

It is noted that none of the prior art suggests any means for effecting a rapid and thorough extraction of a pre-treated pulp in the manufacture of paper-making grades of pulp. Accordingly, it is an object of a broad aspect of this invention to provide an improved rapid and thorough extraction of electrolytes from a pre-treated pulp, in the manufacture of paper-making grades of pulp.

It is an object of a specific aspect of this invention to provide an improved rapid bleaching process which includes a rapid and thorough extraction of the pre-treated pulp, in the manufacture of paper-making grades of pulp, by an alkaline extraction procedure.

An object of another aspect of this invention is the provision of such an extraction technique which is ecologically attractive in its conservation of water.

An object of a further aspect of this invention is the provision of such an extraction technique in which the extracted pulp is delivered at high consistency suitable for fluffing and treating with the gaseous reagent in the next bleaching stage in the manufacture of paper-making grades of pulp.

Statement of the Invention

It has unexpectedly been found that when a pre-treated pulp, e.g. a chlorinated and washed pulp which is to be used in the manufacture of paper-making grades of pulp is mixed rapidly (usually whereby the mixing is completed within about 5 minutes) with an aqueous solution of an alkali, e.g. sodium hydroxide, at a concentration of about 0.5 to 5% by weight, at a mixing consistency of up to about 12%, at a temperature of from about 40°C. to about 90°C., and is substantially immediately thereafter rapidly pressed (i.e. generally whereby the pressing is completed within about one minute) to a consistency of from about 18% to about 40%, then such a single press extraction treatment is as effective as is a conventional alkaline extraction (at about 60°C. to about 80°C.) for about 1 hour, in reducing the Kappa No. of a chlorinated pulp to similar levels.

BRIEF DESCRIPTION OF THE DRAWINGS

In the accompanying drawings,

FIG. 1 is a schematic flow diagram of a conventional prior art alkaline extraction in the manufacture of paper pulp,

FIG. 2 is a schematic flow diagram of a prior art hot caustic purification in the manufacture of dissolving pulp,

FIG. 3 is a schematic flow diagram of a prior art cold caustic purification in the manufacture of dissolving pulp, and

FIG. 4 is a schematic flow diagram of the alkaline extraction of the present invention in the manufacture of paper pulp.

In the conventional alkaline extraction as shown in FIG. 1, the operating conditions vary all the way from 0.5 to 3.0 NaOH on pulp, 40° to 70°C., 60 to 120 min., and 3 to 16% stock consistencies with the higher levels being generally preferred for steam and space econ-

omy. Specifically in FIG. 1 the wood pulp is chlorinated in chlorinator 10 and is subjected to a water wash and thickening in thickener 11. This provides a pulp having a consistency up to 12%.

5 The thickened pulp is mixed in a mixer 12 with steam, water and NaOH, in an amount of about 2 to about 5% based on the pulp. The consistency of the mixture is about 10% and the temperature is generally in the range of 60° - 70°C. The mixture is passed to a reaction tower 13, where the residence time is about 1 to 2 hours. After this time the pulp is washed in a washer 14 and discharged at 15 when the NaOH is substantially completely used up.

15 In hot caustic purification, the NaOH applied to the pulp is generally within the range of 0.5 to 2%, the consistency of the pulp is generally 10 to 22%, the temperature is generally within the range of 50° to 145°C., and the time of treatment is generally from one-fourth to 4 hours. Specifically in FIG. 2 the pulp is chlorinated in chlorinator 20 and is subjected to a wash and thickening in thickener 21. This provides a pulp having a consistency of up to 12%.

25 The thickened pulp is mixed in a mixer 22 with water and NaOH at a maximum concentration of 16% on the pulp. The consistency during the mixing is 12 to 18% and the temperature is 70° to 120°C or even higher. The mixture is passed to a reaction tower 23, where the reaction residence time is 1 or 2 hours or more. The product is then pressed in a press 26 to a consistency of 25 to 35% and is withdrawn at 27. The pressed pulp still contains residual NaOH.

35 In the cold caustic purification the operating conditions may be generally based on the use of 6 to 8% caustic soda solution at 3% consistency for 30 to 60 minutes at 30° to 40°C. Specifically in FIG. 3, the pulp is chlorinated in chlorinator 30 and is subjected to a wash and thickening in thickener 31. This provides a pulp having a consistency of up to 12%.

40 The thickened pulp is mixed in a mixer 32 with water and NaOH at a concentration of 9 to 16% with the pulp at a consistency of 50% at ambient temperature. The mixture is passed to a purifier comprising a plurality of reaction towers 33 and presses 36 so that there are many sequences of the caustic reaction in the reaction tower and the pressing. The total purification time is 1 to 2 hours. The pressed pulp still contains residual NaOH.

50 In the rapid press extraction of the present invention shown in FIG. 4, the pulp is chlorinated in chlorinator 40 and is subjected to a water wash and thickening in thickener 41. This provides a pulp having a consistency up to 12%.

55 The thickened pulp is mixed in a mixer 42 with steam and with 0.5 to 5% NaOH based on the pulp, the mixing taking place at a consistency of less than 12%, preferably 4 to 12%. The temperature is within the range of 40° to 90°C., preferably 60° to 80°C. The mixing is generally completed within about 5 minutes, and no further reaction time is necessary. The mixture is then substantially immediately rapidly pressed in press 46 to a consistency of about 18 to 40%, preferably 20 to 40%. The pressing is generally completed within about 1 minute. The pulp withdrawn at 45 requires little or no water washing and yet has no residual NaOH left thereon.

Other Features of the Invention

In certain aspects of this invention, the pulp is preferably at a consistency of about 4 to about 12% when it is mixed with the alkali, and the mixing temperature is preferably about 60°C. to about 80°C.

In a specific aspect of the invention the alkali is sodium hydroxide and the concentration of NaOH is interrelated to the Kappa Number of the pulp, according to the following table:

PULP	% NaOH on Pulp after chlorination or oxidative treatment
Hardwood Kraft pulp Kappa No. 10 to 20	0.5 - 2
Softwood Kraft pulp Kappa No. 20 to 40	2 - 4
Bisulfite pulp Kappa No. 30 to 50	3 - 5

The pulp is preferably pressed to a consistency of about 20% to 40%. The rapid mixing is generally completed within about 5 minutes, although longer times may be used, and the pressing is generally completed within about 1 minute, although longer times may be used.

By another aspect of this invention, an improved bleaching procedure is provided in the manufacture of paper-making grades of pulp, comprising: (a) subjecting a comminuted pulp to a bleaching treatment; (b) subjecting the bleached pulp to a washing treatment with water; and (c) rapidly mixing the washed pre-bleached pulp at a consistency up to about 12% with an aqueous solution of an alkali at a concentration of 0.5 to 5% based on the pulp, and, substantially immediately thereafter, subjecting such mixture to a rapid press extraction to a consistency of about 18% to about 40%.

In a specific aspect of the invention the alkali is sodium hydroxide and the concentration of NaOH is interrelated to the Kappa Number of the pulp, according to the following table:

PULP	% NaOH on Pulp after chlorination or oxidative treatment
Hardwood Kraft pulp Kappa No. 10 to 20	0.5 - 2
Softwood Kraft pulp Kappa No. 20 to 40	2 - 4
Bisulfite pulp Kappa No. 30 to 50	3 - 5

The pulp is preferably pressed to a consistency of about 20% to 40%. The rapid mixing is generally completed within about 5 minutes, although longer times may be used, and the pressing is generally completed within about 1 minute, although longer times may be used.

In certain aspects of the invention, the bleaching of the comminuted pulp in step (a) above, is carried out: (i) on a pulp pressed to a consistency of about 30% and in fluffed-up form as fibre and fibre aggregates, the bleaching agent being chlorine gas; (ii) on a pulp at a consistency of about 3.5%, the bleaching agent being chlorine in the aqueous phase; or (iii) on a pulp at a consistency of about 12%, the bleaching agent being chloride dioxide.

DESCRIPTION OF PREFERRED EMBODIMENTS

The following examples show unexpected advances over prior art procedures obtained by carrying out the process of aspects of this invention:

Pretreating Procedures

Pretreating Procedure (I)

Unbleached kraft pulp from coniferous wood, characterized by Roe Chlorine No. 5.6, Kappa No. 38.1, and a 0.5% CED viscosity of 24.2 was pressed to 30% consistency. The pulp was comminuted into fibre and fibre aggregates using the apparatus described in U.S. Pat. No. 3,630,828 issued Dec. 28, 1971. The pulp was then treated with chlorine gas for 1 minute (4.5% chlorine on oven dried pulp); the pH of the chlorinated pulp was 1.0. The pulp was then diluted to 1% consistency with deionized water and then brought to 30% consistency by pressing. One form of apparatus which may be used in the rapid pressing procedure of broad aspects of this invention is a hydraulic press, but other types of equipment such as, for example, screw presses or rolls, are equally suitable. The principal elements of one form of press are an upright cylindrical shell having a perforated removable bottom, a close-fitting inner ram, and means of applying loads to the ram. Pressures of the order of 100 pounds per square inch of cross-section are adequate. Ten gram portion samples of the prepared pulp were treated in the following manner.

Pretreating Procedure (II)

Unbleached kraft pulp from coniferous wood, characterized by Roe Chlorine No. 4.6, Kappa No. 26.3, and a 0.5% CED viscosity of 24.6 cps was chlorinated at 3.5% consistency for 1 hour at 25°C. Chlorine consumption was 5.1% chlorine on o.d. pulp. After chlorination, the pulp was washed copiously with deionized water, and brought to 12% consistency over a Buchner funnel covered with a 100 mesh screen. Samples of the prepared pulp were treated in the following manner.

Pretreating Procedure (III)

Unbleached kraft pulp from deciduous wood, characterized by Roe Chlorine No. 1.3, Kappa No. 13.7, and a 0.5% CED viscosity of 20.2 cps, was chlorinated under conventional conditions, 3.5% consistency for 45 minutes at 20°C. Chlorine uptake was 3.0%. After chlorination, the pulp was washed copiously with deionized water and brought to 12% consistency by draining on a Buchner funnel covered with a 100 mesh screen.

Pretreating Procedure (IV)

Unbleached sulphite pulp from coniferous wood, characterized by Roe Chlorine No. 7.6, Kappa No. 38 and 0.5% CED viscosity of 28.2 cps, was pressed to 30% consistency. The pulp was comminuted into fiber and fiber aggregates using the apparatus described in U.S. Pat. No. 3,630,828, issued Dec. 28, 1971. The pulp was then treated with chlorine gas for 1 minute (5.2% chlorine on o.d. pulp); pH of the chlorinated pulp was 0.9. The pulp was then diluted to 1% consistency with deionized water and brought to 30% consistency by pressing.

Pretreating Procedure (V)

A kraft pulp from the southern United States, Roe Chlorine No. 3.8, Kappa No. 21.1, and CED viscosity of 17.0, was chlorinated, washed, extracted, treated with chlorine dioxide, and washed, so that the brightness after these conventional treatments was 82.4. The

pulp was then brought to 12% consistency by draining on a Buchner funnel fitted with a 100 mesh screen.

EXAMPLE 1 — EFFECT OF MULTIPLE PRESSINGS ON PRESS EXTRACTION OF CHLORINATED PULP

Example 1A

One portion of the chlorinated washed pulp from the Pretreating Procedure (I) was extracted with dilute sodium hydroxide (3.5% NaOH o.d. pulp at 10% consistency for 1 hour at 60°C.), and then washed with deionized water at 1% consistency. The washing effluent was removed from the pulp and the colour, BOD and COD measured by appropriate tests. The pulp was then made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table I.

Example 1B

One portion of the treated pulp from the Pretreating Procedure (I) was mixed rapidly (30 seconds) with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 60°C. The consistency of the resulting mixture was 10%. The mixture was immediately pressed to 31% consistency, and this pressing effluent was removed from the pulp and the colour, BOD and COD measured by appropriate tests. The pulp was then washed with deionized water at 1% consistency and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table I.

Example 1C

One portion of the treated pulp from the Pretreating Procedure (I) was mixed rapidly with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 60°C. The consistency of the mixture was 10%. The mixture was immediately pressed to 30% consistency. The effluent from the pressing was quickly remixed a second time, so that the consistency was again 10%, and the pulp repressed immediately to 29%. The pressing effluent was removed from the pulp, the colour, BOD and COD measured by appropriate tests. The pulp was then washed with deionized water at 1% consistency and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table I.

Example 1D

One portion of the treated pulp from the Pretreating Procedure (I) was mixed rapidly with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 60°C. The consistency of the mixture was 10%. The mixture was then immediately pressed to 30% consistency. The pressing effluent was then mixed with the press cake so that the consistency was 10% and immediately pressed again to 30%. The effluent was again mixed with the press cake so that the consistency of mixing was 10%, and then the mixture was pressed immediately to 32%. The pressing effluent was removed from the pulp and the colour, BOD and COD measured by appropriate tests. The pulp was then washed with deionized water

at 1% consistency, and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table I.

Example 1E

One portion of the chlorinated washed pressed pulp from the Pretreating Procedure (I) was extracted with dilute sodium hydroxide (3.5% NaOH on o.d. pulp at 10% consistency for 1 hour at 80°C.), and then washed with deionized water at 1% consistency. The washing effluent was removed from the pulp and the colour, BOD and COD measured by appropriate tests. The pulp was then made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table I.

Example 1F

One portion of the treated pulp from the Pretreating Procedure (I) was mixed rapidly (30 seconds) with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 80°C. The consistency of the resulting mixture was 10%. The mixture was immediately pressed to 20% consistency and this pressing effluent was removed from the pulp and the colour, BOD and COD measured by appropriate tests. The pulp was then washed with deionized water at 1% consistency and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table I.

Example 1G

One portion of the treated pulp from the Pretreating Procedure (I) was mixed rapidly with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 80°C. The consistency of the mixture was 10%. The mixture was immediately pressed to 20% consistency. The effluent from the pressing was quickly remixed a second time, so that the consistency was again 10%, and the pulp repressed immediately to 18%. The pressing effluent was removed from the pulp, the colour, BOD and COD measured by appropriate tests. The pulp was then washed with deionized water at 1% consistency and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table I.

Example 1H

One portion of the treated pulp from the Pretreating Procedure (I) was mixed rapidly with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 80°C. The consistency of the mixture was 10%. The mixture was then immediately pressed to 20% consistency. The pressing effluent was then mixed with the press cake so that the consistency was 10% and immediately pressed again to 20%. The effluent was again mixed with the press cake so that the consistency of mixing was 10%, and then the mixture was pressed immediately to 22%. The pressing effluent was removed from the pulp, and the colour, BOD and COD measured by appropriate tests. The pulp was then washed with deionized water at 1% consistency, and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table I.

TABLE I

EFFECT OF TEMPERATURE AND NUMBER OF PRESSINGS ON KAPPA NO.								
Western Softwood Kraft Pulp, Roe Chlorine No., 5.6, Kappa No. 38.1, 0.5% CED Viscosity, cps., 24.2 Pentosans 6.7% Alpha-cellulose 89.2%*								
Example No.	NaOH % on o.d. pulp	Temp. °C.	Pulp During Mixing	Alpha-cellulose Consistency% After Extraction	pH After Extraction	Extraction Time mins.	Pulp Properties After Washing Kappa No.	0.5% CED Viscosity
1A	3.5	60	10	—	10.5	—	7.9	24.6
1B	3.5	60	10	31	10.9	0.75	7.9	25.0
1C	3.5	60	10	29	10.5	0.75	7.5	26.3
1D	3.5	60	10	32	10.1	0.75	7.5	25.7
1E	3.5	80	10	—	10.3	0.75	7.5	25.2
1F	3.5	80	10	20	10.6	0.75	7.2	24.1
1G	3.5	80	10	18	10.3	0.75	7.1	24.8
1H	3.5	80	10	22	10.0	0.75	6.3	24.3
Example No.	Pulp Properties After Washing		Effluent Properties		COD	BOD	U.S. Gal H ₂ O Removed/ton of pulp	
	Pentosans %	Alpha-Cellulose %	Volume Removed mls	Colour APHA units Mg/pt/l 465 Mu	ppm	ppm		
1A	6.7	90.3	917	3,028	589	115	22,000	
1B	6.8	88.9	68	33,216	5870	1430	1,640	
1C	6.7	90.1	65	35,693	6325	1500	1,570	
1D	6.7	90.2	69	36,170	7388	1550	1,665	
1E	6.6	90.4	917	3,121	618	115	22,000	
1F	6.7	89.2	51	33,248	6128	1500	1,225	
1G	6.7	89.9	45	43,211	6692	1550	1,080	
1H	6.7	90.2	55	48,327	8683	2050	1,325	

* after acid chloride treatment

As seen in Table I, a significant advantage in the press extraction of the chlorinated pulp appears to be that a rapid, preferably single press extraction (i.e. taking a time, generally, of less than 1 minute) can reduce the Kappa number of a chlorinated pulp to a value which is similar to that accomplished by conventional treatment with the same quantity of caustic, at the same temperature, but for a much shorter time than the conventional time period of 60 minutes. The results in Examples 1B, 1C and 1D show that two and three press extractions do not greatly reduce the Kappa number from that obtained after the first press extraction, and this is an unexpected result. It should be noted that the loadings of the effluent, as expressed in colour units, COD units and BOD units are larger for the press extracted pulp; this means that a greater concentration of impurities will be captured in a smaller volume of water, increasing the concentration of such impurities so that the removal or purification task may be much simpler.

EXAMPLE 2 — EFFECT OF MIXING CONSISTENCIES ON PRESS EXTRACTION OF CHLORINATED PULP

The following examples are intended to show the effect on reduction of Kappa number by mixing chlorinated and washed pulp with dilute solutions of sodium hydroxides at different mixing consistencies (5 and 10%) and thereupon immediately pressing the mixture by a single pressing stage to consistencies of from 20 to 40%.

Example 2A

One portion of the chlorinated washed pressed pulp from the Pretreating Procedure (I) was mixed rapidly (2 minutes) with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 60°C. The consistency of the resulting mixture was 5%. The mixture was immediately pressed to 21% consistency in 0.39 minute, and this pressing effluent was removed from the pulp.

The pulp was then washed with deionized water at 1% consistency, and made into handsheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table II.

Example 2B

One portion of the chlorinated washed pressed pulp from the Pretreating Procedure (I) was mixed rapidly (2 minutes) with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 60°C. The mixture was pressed to 31% consistency in 0.52 minutes and the pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency, and made into handsheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table II.

Example 2C

One portion of the chlorinated washed pressed pulp from the Pretreating Procedure (I) was mixed rapidly (two minutes) with an aqueous solution of sodium hydroxide (3.5% on o.d. pulp) at 60°C. The mixture was immediately pressed to 38% consistency (pressing time 1.15 minutes), and the pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency, and made into handsheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table II.

Example 2D

One portion of the chlorinated washed pressed pulp from the Pretreating Procedure (I) was mixed rapidly (2 minutes) with an aqueous solution of sodium hydroxide (10% on o.d. pulp) at 60°C. The mixture was immediately pressed to 22% consistency (pressing time 0.33 minutes), and the pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency, and made into hand-

sheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table II.

Example 2E

One portion of the chlorinated washed pressed pulp from the Pretreating Procedure (I) was mixed rapidly (2 minutes) with an aqueous solution of sodium hydroxide (10% on o.d. pulp) at 60°C. The mixture was immediately pressed to 30% consistency (pressing time 0.45 minute), and the pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency, and made into handsheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table II.

Example 2F

One portion of the chlorinated washed pressed pulp from the Pretreating Procedure (I) was mixed rapidly (two minutes) with an aqueous solution of sodium hydroxide (10% on o.d. pulp) at 60°C. The mixture was immediately pressed to 38% consistency (pressing time 1.0 minute), and the pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency, and made into handsheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table II.

TABLE II

EFFECT OF MIXING AND PRESSING CONSISTENCY DURING AKALINE PRESS EXTRACTION						
Softwood Kraft Pulp, Roe Chlorine No. 5.6, Kappa No., 38.1; 0.5% CED Viscosity, cps. 24.2 Pentosans 6.7% Alpha-cellulose 89.2%						
Example No.	NaOH % on o.d. pulp	Temp. °C.	Pulp Consistency		After Extraction	Extraction Time Mins.
			During Mixing	After Extraction		
2A	3.5	60	5	21	11.2	.39
2B	3.5	60	5	31	11.1	.52
2C	3.5	60	5	38	10.8	1.0
2D	3.1	60	10	22	11.0	.33
2E	3.5	60	10	30	10.9	.45
2F	3.5	60	10	38	10.7	1.0
Example No.			Pulp Properties After Washing		Alpha-cellulose %	
	Kappa No.	0.5% CED Viscosity cps	Pentosans %			
2A	7.9	25.0	6.8		88.9	
2B	7.7	24.7	6.8		88.7	
2C	7.4	24.9	6.7		89.2	
2D	8.2	24.8	6.8		89.6	
2E	7.9	25.1	6.7		89.9	
2F	7.4	24.9	6.7		89.9	

*after acid chloride treatment

As seen in Table II, there was very little effect on the Kappa number when the pulp consistency during mixing is within the range of 5 - 10% or when the pulp consistency after pressing is within the range 21 - 38%.

EXAMPLE 3 — EFFECT OF CONVENTIONAL CHLORINATION ON PRESS EXTRACTION OF CHLORINATED PULP

The following example is intended to show the use of

the procedure of this invention on a pulp which has been chlorinated by conventional aqueous treatment.

Example 3A

One portion of the chlorinated washed pulp from the Pretreating Procedure (II) was extracted with dilute sodium hydroxide (2.0% NaOH on o.d. pulp at 10% consistency for one hour at 60°C.), and then washed with deionized water at 1%. The pulp was then made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table III.

Example 3B

One portion of the treated pulp from the Pretreating Procedure (II) was mixed rapidly (3 minutes) with an aqueous solution of sodium hydroxide (2.0% on o.d. pulp at 60°C.). The consistency of the resulting mixture was 10%. The mixture was immediately pressed to 30% consistency, and the effluent removed from the pulp. The pulp was then washed with deionized water at 1% consistency and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table III.

Example 3C

One portion of the treated pulp from the Pretreating Procedure (II) was mixed rapidly (2 minutes) with an

aqueous solution of sodium hydroxide (2.0% NaOH on o.d. pulp) at 60°C. The consistency of the mixture was 10%. The mixture was immediately pressed to 30% consistency. The effluent from the pressing was quickly remixed a second time, so that the consistency was again 10%, and the pulp repressed immediately to 30%. The pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table III.

TABLE III

Eastern Softwood Kraft, Roe Chlorine No., 4.6; Kappa No. 26.3 0.5% CED Viscosity No., 24.6 cps; Conventional Chlorination, 5.1% Cl on o.d. Pulp Pentosans 9.9%; Alpha-cellulose 87.5%*						
Example No.	NaOH % on o.d pulp	Temp. °C.	Pulp Consistency During Mixing	Pulp Consistency After Extraction	pH After Extraction	Extraction Time mins.
3A	2.0	60	10	—	11.3	—
3B	2.0	60	10	30	11.5	0.75
3C	2.0	60	10	30	11.3	0.75
Example No.	Pulp Properties After Washing					
	Kappa No.	0.5% CED Viscosity cps	Pentosans %	Alpha-cellulose %		
3A	7.1	24.0	8.8	87.6		
3B	7.1	24.2	8.9	87.2		
3C	6.3	24.1	8.6	87.8		

*after acid chloride treatment

As shown in Table III, a significant advantage in the press extraction of a commercially-chlorinated pulp appears to be that a single such press extraction can reduce the Kappa number of a chlorinated pulp to a value similar to that which can be accomplished by conventional treatment with the same quantity of caustic, at the same temperature, but for a much shorter time than the conventional time period of 60 minutes. The results in Examples 3B and 3C show that two press extractions did not greatly reduce the Kappa number from that achieved by the first extraction and this is an unexpected result.

Example 4B

One portion of the chlorinated washed pulp from the Pretreating Procedure (III) was mixed rapidly (3 minutes) with an aqueous solution of sodium hydroxide, 2.0% on o.d. pulp, at 60°C. The consistency of the mixture was 10%. The mixture was immediately pressed to 30% consistency and the pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency, and made into handsheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table IV.

TABLE IV

Eastern Hardwood Kraft, Roe Chlorine No. 1.3; Kappa No., 13.7; 0.5% CED Viscosity, 20.2 cps Conventional Chlorination 3.0% Cl ₂ on o.d. Pulp Pentosans 22.3%; Alpha-cellulose 86.9%*						
Example No.	NaOH % on o.d. pulp	Temp. °C.	Pulp Consistency During Mixing	Pulp Consistency After Extraction	pH After Extraction	Extraction Time mins.
4A	2.0	60	10	—	11.2	—
4B	2.0	60	10	30	11.3	0.50
Example No.	Pulp Properties After Washing					
	Kappa No.	0.5% CED Viscosity cps	Pentosans %	Alpha-cellulose %		
4A	4.8	18.6	21.7	86.8		
4B	4.9	18.8	21.9	86.6		

*after acid chloride treatment

EXAMPLE 4 — EFFECT OF CONVENTIONAL CHLORINATION ON PRESS EXTRACTION OF CHLORINATED PULP

Example 4A

One portion of the chlorinated and washed pulp from the Pretreating Procedure (III) was extracted with dilute sodium hydroxide (2.0% NaOH on o.d. pulp at 10% consistency for 1 hour at 60°C.) and then washed with deionized water at 1%. The pulp was then made into handsheets and Kappa number and CED viscosity measured.

The results obtained are shown in Table IV.

As shown in Table IV, the single press extraction of this invention has significant utility on conventionally chlorinated pulp.

EXAMPLE 5 — EFFECT OF GAS PHASE CHLORINATION OF SINGLE PRESS EXTRACTION

Example 5A

One portion of the chlorinated washed pressed pulp from the Pretreating Procedure (IV) was extracted with dilute sodium hydroxide (2.5% NaOH on o.d. pulp at 10% consistency for one hour at 60°C.), and then

washed with deionized water at 1% consistency. Pulp was then made into handsheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table V.

Example 5B

One portion of the treated pulp from the Pretreating Procedure (IV) was mixed rapidly (30 seconds) with an aqueous solution of sodium hydroxide (2.5% on o.d. pulp), at 60°C. The consistency of the resulting mixture was 10%. The mixture was immediately pressed and this pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency and made into handsheets and the Kappa number and CED viscosity measured.

The results obtained are shown in Table V.

Example 5C

One portion of the treated pulp from the Pretreating Procedure (IV) was mixed rapidly (30 seconds) with an aqueous solution of sodium hydroxide (2.5% on o.d. pulp) at 60°C. The consistency of the mixture was 10%. The mixture was immediately pressed to 30% consistency. The effluent from the pressing was quickly re-mixed a second time, so that the consistency was again 10%, and the pulp repressed immediately to 30% consistency, and the pressing effluent was removed from the pulp. The pulp was then washed with deionized water at 1% consistency, and made into handsheets, and the Kappa number and CED viscosity measured.

The results obtained are shown in Table V.

TABLE V

Example No.	NaOH % on o.d. pulp	Temp. °C.	Pulp Consistency		pH After Extraction	Extraction Time mins.
			During Mixing	After Extraction		
5A	2.5	60	10	—	10.5	—
5B	2.5	60	10	30	10.9	0.75
5C	2.5	60	10	30	10.5	0.75
Example No.	Kappa No.	Pulp Properties After Washing		Alpha-cellulose %		
		0.5% CED Viscosity cps	Pentosans %			
5A	1.2	28.0	6.7	87.2		
5B	1.5	28.1	7.0	86.9		
5C	1.3	28.0	6.8	87.0		

*after acid chloride treatment

As seen in Table V, the single press extraction of this invention has significant utility on pulp which has been subjected to gas-phase chlorination.

EXAMPLE 6

The following examples are intended to show that an extraction or neutralization can be carried out by the alkaline press extraction technique, with or without a washing stage, following the neutralization or extraction stage in a multi-stage process, between each of the two oxidation stages, such as, for example, chlorine dioxide, chlorine dioxide (DD), or hypochlorite, chlorine dioxide (HD), in which a neutralization or extraction is usually carried out to improve the brightness of the last oxidation stage.

Example 6A

One portion of the semi-bleached pulp from the Pretreating Procedure (V) was extracted with dilute sodium hydroxide (1.0% NaOH on o.d. pulp at 10% consistency for 1 hour at 60°C.), and then washed with deionized water at 1% consistency. The pulp was then brought to 12% consistency over a Buchner funnel, and then bleached with 0.5% chlorine dioxide (on o.d. pulp for 3 hours at 70°C.). The pulp was then washed and made into handsheets, and the resulting brightness was 91.1 on the Elrepho brightness meter.

Example 6B

One portion of the semi-bleached pulp from the Pretreating Procedure (V) was mixed rapidly (2 minutes) with an aqueous solution of sodium hydroxide (1.0% on o.d. pulp at 60°C.). The consistency of the resulting mixture was 10%. The mixture was immediately pressed to 30% consistency, and the pressing effluent was removed from the pulp. Without any further washing, the pulp was mixed with chlorine dioxide solution (0.5% chlorine dioxide on o.d. pulp) at a consistency of 10% for 3 hours at 70°C. The pulp was then washed, and made into handsheets for brightness determination. The brightness was 90.8 on the Elrepho brightness meter.

Example 6C

One portion of the semi-bleached pulp from the Pretreating Procedure (V) was mixed rapidly with an aqueous solution of sodium hydroxide (1.0% on o.d.

pulp) at 60°C. The consistency of the mixture was 10%. The mixture was then immediately pressed to 30% consistency, and the pressing effluent removed from the pulp. The pulp was then washed with deionized water at 1% consistency and then brought to 12% consistency by a Buchner funnel fitted with a 100 mesh screen. The pulp was then mixed with chlorine dioxide solution (0.5% chlorine dioxide on o.d. pulp at 10% consistency for 3 hours at 70°C.). After washing, the pulp was made into handsheets and the brightness measured. The brightness was 91.1 on the Elrepho brightness meter.

As seen above, it is clear that an alkaline press extraction on a pulp between two oxidizing stages does not need any further washing after the press extraction treatment of the present invention.

While the present invention has been directed to the use of sodium hydroxide as the alkali extraction agent in the Examples, it is possible to perform the alkali extraction with calcium hydroxide or with sodium bicarbonate with equivalent improvement over prior art extraction procedures using those alkali agents.

The preceding examples can be repeated with similar success by substituting the generically and specifically described reactants and operating conditions of this invention for those used in the preceding examples.

From the foregoing description, one skilled in the art can easily ascertain the essential characteristics of this invention, and without departing from the spirit and scope thereof, can make various changes and modifications of the invention to adapt it to various usages and conditions. Consequently, such changes and modifications are properly, equitably, and "intended" to be, within the full range of equivalence of the following claims.

I claim:

1. An improved alkaline extraction procedure in the manufacture of paper-making pulp, which comprises:

- a. mixing a pretreated paper-making grade of pulp, having been pretreated with a bleaching chemical and then washed, at a consistency of up to about 12% with an aqueous solution of an alkali of a concentration of 0.5 - 5% based on the pulp and at a temperature of about 40° to about 90°C., and then without an intervening washing step;
- b. substantially immediately thereafter subjecting said mixture to a rapid press extraction, during which no substantial washing occurs, for a sufficient time and under a sufficient pressure to obtain a consistency of about 18% to about 40%, the total treatment time of steps (a) and (b) being for a time sufficient to obtain a resultant extracted pulp containing less than 91% α -cellulose, and retaining a substantial quantity of hemi-celluloses, a substantial degree of the extraction being accomplished in said rapid press extraction.

2. The process of claim 1 wherein the alkali is NaOH, and in which the concentration thereof is interrelated to the Kappa number of the pulp according to the following table.

PULP	% NaOH on PULP after chlorination or Oxidative Treatment
Hardwood Kraft pulp Kappa No., 10 to 20	0.5 - 2
Softwood Kraft pulp Kappa No., 20 - 40	2 - 4
Bisulfite pulp Kappa No., 30 - 50	3 - 5

3. The process of claim 1 wherein the mixing step is substantially completed within about 5 minutes and wherein the rapid press extraction is a single extraction step which is substantially completed within about 1 minute.

4. The process of claim 1 wherein the consistency of the pulp at the mixing step is about 4 to about 12%.

5. The process of claim 1 wherein the temperature during the mixing step is about 60°C. to about 80°C.

6. An improved bleaching procedure in the manufacture of paper-making pulp, comprising:

- a. subjecting a comminuted pulp to a bleaching treatment;
- b. subjecting said bleached pulp to a washing treatment with water;

c. mixing said washed bleached pulp at a consistency of up to about 12% with an aqueous solution of an alkali of a concentration of 0.5 - 5% based on the pulp and at a temperature of about 40° to about 90°C., and then without an intervening washing step;

d. substantially immediately thereafter subjecting said mixture to a rapid press extraction, during which no substantial washing occurs, for a sufficient time and under a sufficient pressure to obtain a consistency of about 18% to about 40%, the total treatment time of steps (c) and (d) being for a time sufficient to obtain a resultant extracted pulp containing less than 91% α -cellulose, and retaining a substantial quantity of hemi-celluloses, a substantial degree of the extraction being accomplished in said rapid press extraction.

7. The process of claim 6 wherein the alkali is NaOH, and in which the concentration thereof is interrelated to the Kappa number of the pulp according to the following table.

PULP	% NaOH on PULP after chlorination or Oxidative Treatment
Hardwood Kraft pulp Kappa No. 10 to 20	0.5 - 2
Softwood Kraft pulp Kappa No. 20 - 40	2 - 4
Bisulfite pulp Kappa No. 30 - 50	3 - 5

8. The process of claim 6 wherein the mixing step is substantially completed within about 5 minutes and wherein the rapid press extraction is a single extraction step which is substantially completed within about 1 minute.

9. The process of claim 6 wherein the consistency of the pulp at the mixing step is about 4 to about 12%.

10. The process of claim 6 wherein the temperature during the mixing step is about 60° C. to about 80° C.

11. The process of claim 6 wherein the bleaching is carried out on a pulp pressed to a consistency of about 30% and in fluffed-up form as fibre and fibre aggregates, the bleaching agent being chlorine gas.

12. The process of claim 6 wherein the bleaching is carried out on a pulp at a consistency of about 3.5%, the bleaching agent being chlorine in the aqueous phase.

13. The process of claim 6 wherein the bleaching is carried out on a pulp at a consistency of about 12%.

14. The process of claim 6, as a multi-stage process, said rapid press extraction being carried out between two oxidation stages, an oxidizing agent being employed in each of said two oxidation stages.

15. The process of claim 14, wherein no water washing stage is carried out between said press extraction stage and the subsequent oxidation stage.

16. The process of claim 14 wherein the oxidizing agent for both stages comprises chlorine dioxide.

17. The process of claim 1 wherein the total treatment time of steps (a) and (b) is substantially less than 1 hour.

18. The process of claim 1, wherein steps (a) and (b) are completed within about 6 minutes.

19. The process of claim 6, wherein the total treatment time of steps (c) and (d) is substantially less than 1 hour.

20. The process of claim 6, wherein steps (c) and (d) are completed within about 6 minutes.