Oct. 14, 1952 P. H. SCHLOSSER ET AL 2,614,102

STEEPING OF CELLULOSE

Filed Feb. 23, 1950 4 Sheets-Sheet 1

FIG. 1

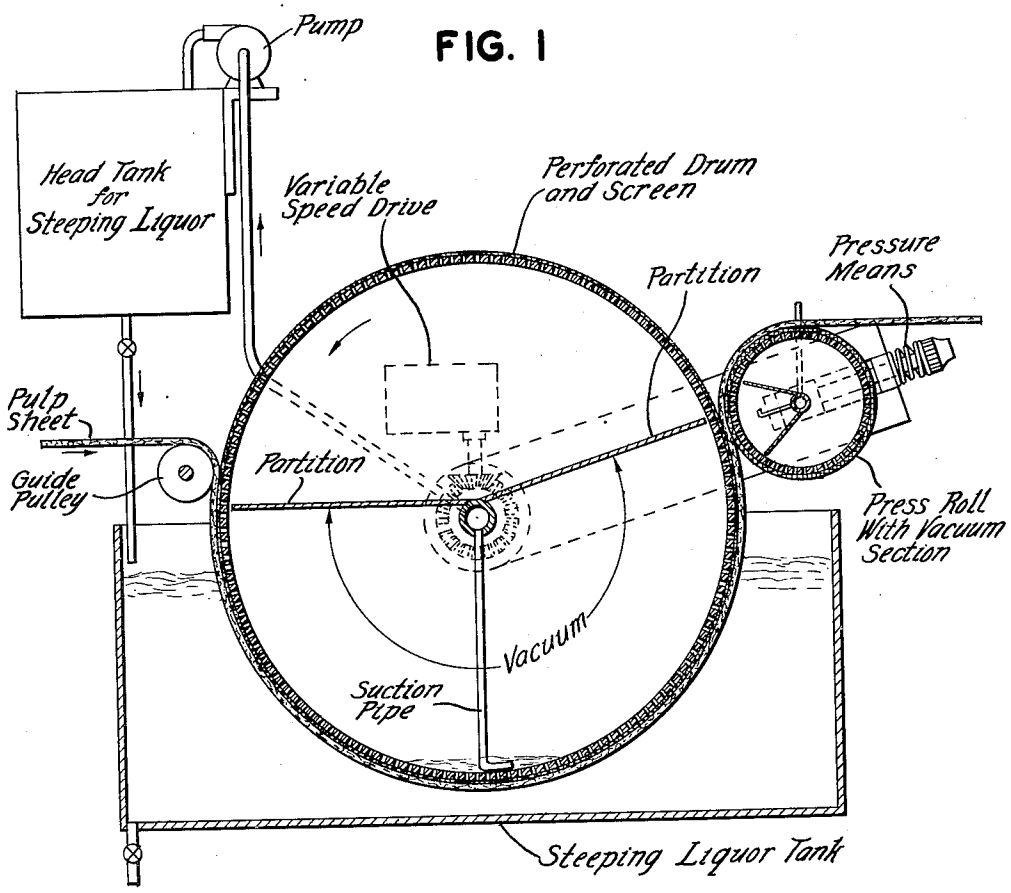
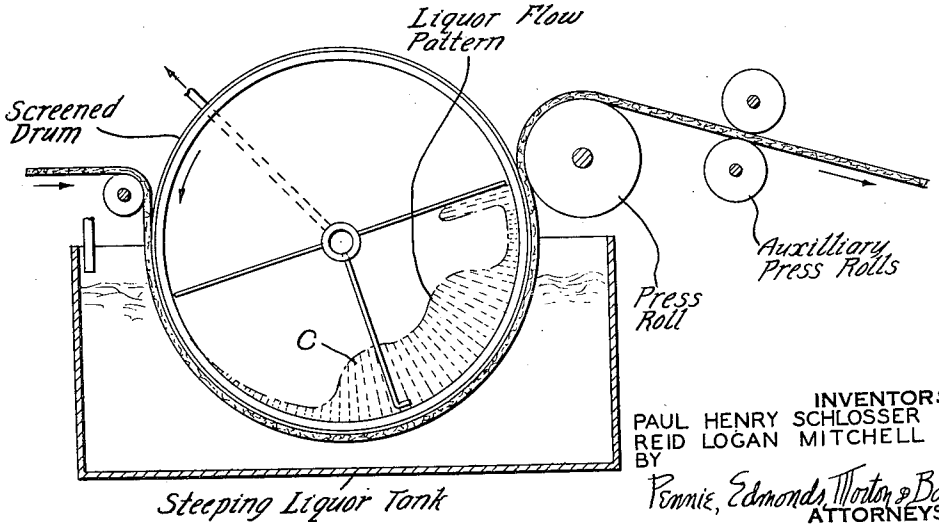


FIG. 2



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FIG. 3

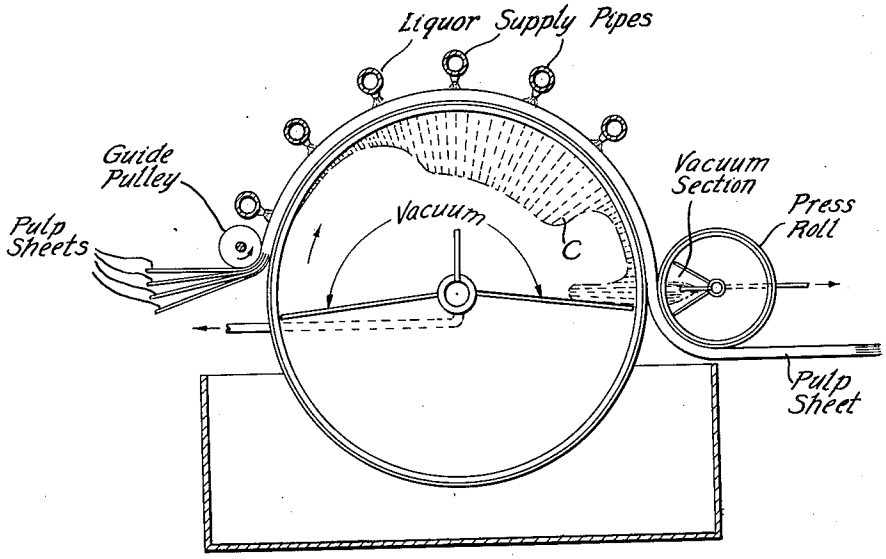


FIG. 4

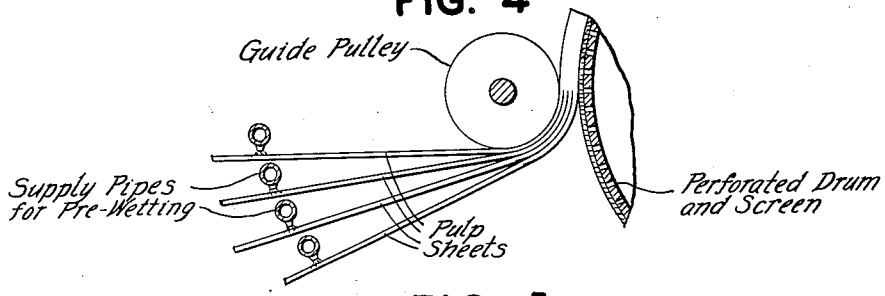
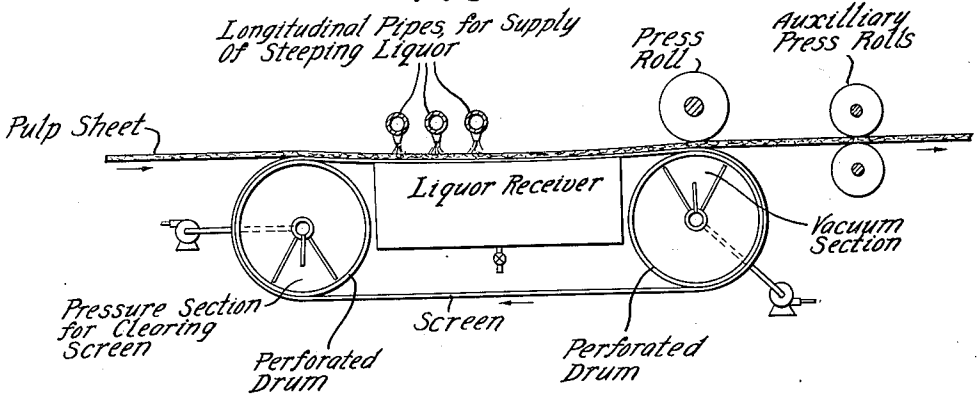


FIG. 5



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STEEPING OF CELLULOSE

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FIG. 6

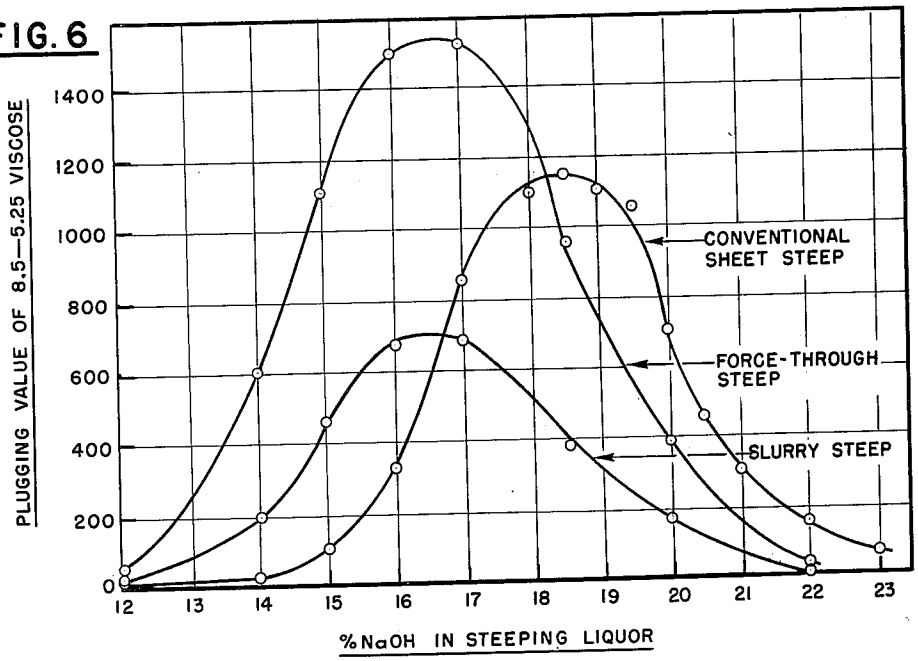
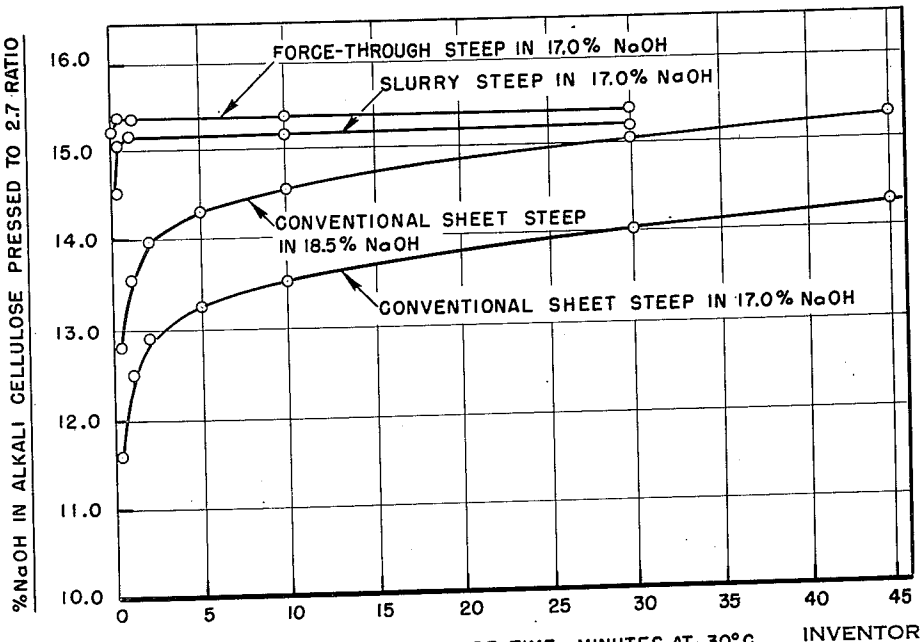


FIG. 7



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STEEPING OF CELLULOSE

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FIG. 8

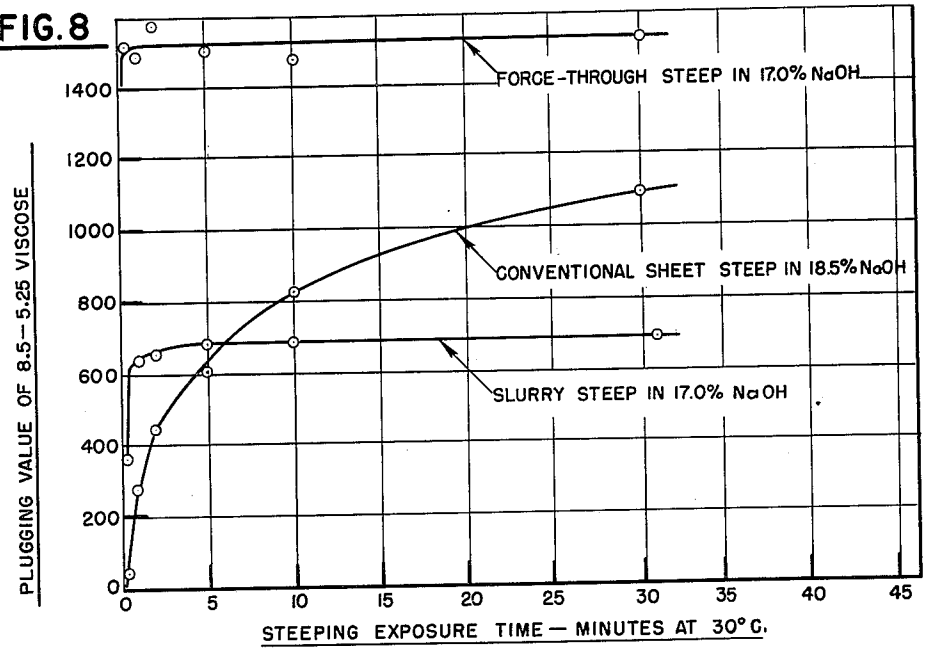
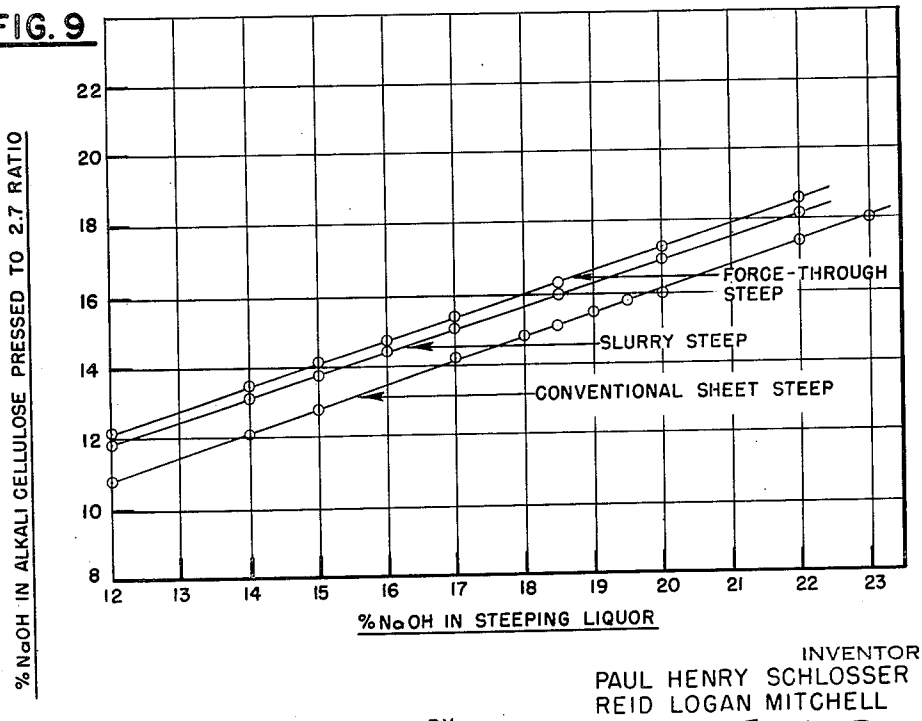


FIG. 9



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UNITED STATES PATENT OFFICE

2,614,102

STEEPING OF CELLULOSE

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Application February 23, 1950, Serial No. 145,676

1 Claim. (Cl. 260—233)

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This invention relates to the steeping of cellulose pulp of the type known as dissolving pulp with a caustic soda solution and has for its object the provision of an improved method of steeping pulp sheets of indefinite length with a caustic soda solution to form alkali cellulose. Our invention aims to increase the efficiency of producing alkali cellulose, and also to produce an alkali cellulose having improved properties.

The sheets of pulp treated according to the invention may be wet or dry and of any desired alpha cellulose content and are usually provided in large rolls which are unwound as the sheets are fed through the operation. Hereinafter, for simplicity, we shall refer to the caustic soda steeping liquor (aqueous solution of NaOH) as "steeping liquor" or simply "liquor."

In accordance with our invention, we pass a continuously moving sheet of cellulose pulp through an operation in which the steeping liquor is forced through the sheet with the attainment of NaOH equilibrium in an amazingly short time and with the use of an unexpectedly small amount of steeping liquor. Moreover, the invention produces a greatly improved alkali cellulose characterized by better uniformity, better shredding properties, and, when converted into viscose, better filtering rates. The invention is advantageously applicable to the viscose process and may, accordingly, be employed as one of the important steps in that process.

In one of its preferred embodiments, the invention comprises forcing steeping liquor through a continuously moving sheet of pulp, then removing the excess steeping liquor, as by vacuum or pressure, during a relatively brief period requiring but seconds. The liquor may be forced through the sheet by sucking it through with a vacuum, or it may be forced through by applying pressure to the liquor. In either means, the liquor flows through the pulp uniformly without becoming depleted in NaOH to any appreciable extent. In other words, all the fibers of the pulp are treated the same period and with the same caustic concentration. Advantageously, we pass the sheet in one continuous movement through one stage of treatment in which a steeping liquor, preferably of relatively low NaOH concentration, is forced through the sheet, and then through another stage of treatment in which the excess steeping liquor is removed, preferably by vacuum or in combination with press rolls or the like.

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For example, the forcing of the liquor through the sheet may be accomplished in from 2 to 60 seconds, 20 seconds usually being an adequate time. Proceeding immediately from the forcing operation, the adhering liquor is removed, preferably by vacuum, to the desired degree of dryness in but a few seconds, say, from 2 to 4 seconds. In sucking out the liquor, the arrangement of fibers is left more or less intact and the sheet is susceptible to efficient shredding. While we may use press rolls in combination with the suction to remove liquor, we prefer not to use such rolling pressure as to disturb the continuity of the sheet and thus impede shredding.

We have confirmed by extensive investigations our conception that effective conversion of the cellulose to alkali cellulose can be accomplished by contacting all the fibers unformly with liquor of uniform NaOH concentration in but a matter of seconds, thus eliminating the relatively long soaking period in which the sheet is left in a body of steeping liquor. The whole operation of forcing the solution through the sheet and removing the excess to a desired dry state may be accomplished in less than a minute, say, from 4 to 20 seconds.

The following is a brief discussion of various conventional methods of producing commercially usable alkali cellulose:

Sheet steeping.—In this operation, pulp sheets are soaked in a tank of steeping liquor; books of pulp sheets are loaded into the sections of a steeping tank, steeping liquor is run in from the bottom at a prescribed rate, the sheets are allowed to soak for a prescribed time, then drained and pressed, by advance of a ram, to the desired ratio of original weight to steeped weight. Under optimum conditions and with good-steeping pulp, this method will produce alkali cellulose of good subsequent processability.

One objection to this method is that it is a batch operation. Another lies in the inherent non-uniformity of NaOH distribution obtained by soaking sheets in books. Even if the steeping liquor penetrates to each sheet surface within a book, there is, nevertheless, a differential in NaOH concentration between the sheet surface and interior. This arises because of the selective adsorption of NaOH from the liquor by the cellulose as the liquor penetrates into the sheet. In practice, this sheet effect is often exaggerated into a book effect by crowding and the liquor is forced to penetrate through several

sheet thicknesses. Liquor reaching the center of the book is depleted in NaOH content while being enhanced in hemi content and in temperature. The liquor reduced in NaOH content reaching the center of books gives inadequate mercerization and results in poor subsequent processability and poor viscose filterability. One good feature, however, of this method of steeping is the desirable physical structure of the steeped and pressed alkali cellulose sheets which are easily shred into an open-structured crumb showing good subsequent reactivity and processability.

Continuous sheet steeping.—In one endeavor to make the steeping process a continuous operation, a continuous sheet of pulp is led from a roll through a tank of steeping liquor and out through a pair of squeeze rolls. This process involves a soaking operation similar to conventional steeping except that the book effect and batch processing are eliminated. It is still subject to similar criticism with respect to non-uniformity of NaOH distribution within the sheet. There are, furthermore, several operating difficulties that restrict its application, namely, (1) the slow rate of wetting and penetration requires a long period of contact with steeping liquor which is obtainable only by excessively slow speed or an undesirably long tank, and (2) the highly swollen sheet, slippery with excess liquor, gives much trouble in handling and pressing on its exit from the soaking tank.

Slurry steeping.—Pulp sheets are dispersed in steeping liquor to give a slurry of fibers which is continuously fed to a pair of perforated rolls on which the fibers are collected and later expressed between the rolls as a mat of pressed alkali cellulose. This method of steeping naturally gives uniform alkalinity among the pulp fibers because each fiber has access to a large volume of steeping liquor. However, in spite of the near-optimum wetting phase, this process does not produce alkali cellulose with the high degree of processability that one would suppose. The disadvantage lies in certain inherent poor-shredding properties of the alkali cellulose which results from the pressing of the mat formed from the caustic slurry. Instead of the fibrous, open, highly porous, finely divided crumb that one customarily obtains by shredding conventionally steeped alkali cellulose from pre-formed sheets, one obtains from slurry steeped alkali cellulose a flaky, soapy, dense crumb of poor porosity, reactivity and processability. Although the chemical uniformity of the slurry wetting phase is near-ideal, it, nevertheless, fails to provide a product which can be handled in subsequent operations to result in highly satisfactory viscose filterability.

In the method of the invention, steeping liquor is continuously forced through a moving pulp sheet as by means of applied vacuum. It is also possible to force the steeping liquor through the pulp sheet by means of applied pressure. The continuous operation requires an extremely short time and the passage of a surprisingly small volume of liquor. The actual time required to force the liquor through the sheet may be accomplished in times as short as 4 seconds. Nevertheless, the wetting phase of this method has proved to be even better than that provided by slurry steeping. In effect, it is substantially equivalent to single fiber steeping in a liquor that does not become depleted by adsorption of NaOH on the fibers. Also by maintaining the preformed sheet structure through the wetting phase, we are able to press and shred a near-ideal structural form which, in addition, is uniformly impregnated, alkalized, and mercerized with NaOH.

Use of suitable means to force the steeping liquor through the sheet is found to give not only a remarkable increase in rate of wetting and penetration but, when vacuum is used in connection with the operation to remove the excess liquor, it further provides a sucked sheet at the end of steeping which is notably easy to handle and press. Both of these features are marked improvements over the continuous soaking of a sheet. It is also surprising to find the NaOH adsorption to reach equilibrium at such a high level in such a short time with such little liquor.

Unlike conventional steeping, the steeping method of the invention provides equivalent uniformity in alkalinity for thick sheets as well as for thin ones. Also, the time for forcing the steeping liquor through the sheet does not increase very much for large increases in thickness of sheet or for increases in volume of liquor forced through. These features are all favorable to the use of high capacity equipment. (See tables and Figs. 6 to 9.)

The slightly higher NaOH equilibrium obtained for a given concentration of steeping liquor by our method over slurry steeping is logically explainable on the basis that in our method there is continual replacement of depleted liquor by successive portions of full-strength liquor while in slurry steeping there is a slight depletion in NaOH of the entire bulk of the liquor surrounding the fibers.

There is a decided advantage in the shredding of the sheets treated according to the invention because the sheet structure is substantially preserved.

Figs. 1 to 5 of the accompanying drawings illustrate diagrammatically arrangements of apparatus suitable for practicing the invention.

The apparatus illustrated in Fig. 1 comprises a perforated and screen covered drum having a vacuum section formed inside the sealing partitions. The perforated drum and screen are immersed in a body of steeping liquor in the tank which is sucked through the sheet and screen and into the space. The liquor which enters the vacuum space is removed through the suction pipe by the pump and is discharged into the head tank from which it may return to the tank. The partitions and suction pipe are, of course, held stationary. The perforated drum and screen may be rotated at any desired speed by the variable speed drive to give the desired retention time during which the steeping liquor is forced through the sheet. As the drum rotates, the sheet of pulp from a roll (not shown) passes over the guide pulley and then over the lower half of the drum. Opposite the vacuum section of the drum, the sheet passes over a perforated press roll having a vacuum section and the sheet is accordingly under a vacuum on both sides at the point of contact with the drum and roll. By regulating the speed of the drum, the time during which the liquor is being sucked through the sheet can be varied. The sheet may be directed to any one of several stages in conversion to viscose.

Fig. 2 illustrates a rotatable screened drum having a vacuum section similar to that of Fig. 1, which drum is also immersed in a body of steeping liquor. In this modification, the press roll is not perforated and auxiliary press rolls are used. The shaded area within the curve C illus-

trates the magnitude of liquor flowing through the sheet. The sudden increase in flow of liquor near the upper right-hand portion of the vacuum section is due to the squeezing of liquor out of the sheet by the press roll simultaneously with the application of vacuum. The auxiliary press rolls may be resorted to for the purpose of effecting a more complete elimination of steeping liquor. Any desired number of separate sheets of pulp may be introduced to the perforated drum and superposed one over the other as they rotate with the drum.

The modified form of apparatus illustrated in Fig. 3 comprises a rotatable perforated drum, as in Fig. 1, but the vacuum section is reversed to the upper position and the steeping liquor is sprayed on or otherwise applied to the pulp sheet by a number of longitudinal supply pipes which extend transversely across the width of the sheet. As the liquor contacts the sheet, it is sucked therethrough and the amount of flow through the sheet at different portions of the drum is indicated by the shaded area within the curve C. It will be noted that there is a very appreciable rate of flow of liquor through the pulp at the point of contact with the press roll having a vacuum section at the point of contact. Several separate sheets of pulp are led to the guide roll where they become superposed into a single sheet in passing over the perforated drum.

Fig. 4 illustrates a modification of the arrangement illustrated in Fig. 3 in which the several longitudinal supply pipes are arranged to direct the steeping liquor onto each sheet individually and prior to its engagement with the perforated drum.

The arrangement of apparatus illustrated in Fig. 5 comprises two spaced perforated drums, one having a vacuum section and the other a pressure section and over which the screen as a continuous belt travels in the direction of the arrow. The liquor receiver has an open grid for a top over which the screen travels and is supported. The steeping liquor is sprayed on or otherwise applied to the pulp sheet by the longitudinal supply pipes. The caustic soda solution may be forced through the sheet under pressure. The solution may also be forced through the sheet by using a vacuum in the liquor receiver to suck the liquor through the sheet. A combination of both pressure on the solution and vacuum in the liquor receiver may be used. The sheet completely impregnated with the steeping liquor passes under the press roll which bears on the screen passing over the vacuum section of the last drum. The liquor is both squeezed and sucked out of the pulp sheet. The auxiliary press rolls may be used to effect a further diminution in the steeping liquor remaining in the pulp sheet. In order to keep the mesh of the screen open so that it will function uniformly over its entire area, one drum is provided with a pressure section and air under pressure is forced through the screen to remove interfering pulp fibers.

Any suitable arrangement of apparatus may be used to practice the invention which will force the steeping liquor to flow through the entire sheet of pulp. This force may be that resulting from the pressure of the atmosphere in those instances where one side of the sheet is placed under a vacuum, or it may be due to the pressure applied to the liquor itself, or a combination of both. The term "force-through," as used herein, does not mean a force such as that resulting from capillary action as when the pulp is merely immersed in or wet with the liquor.

The following are typical examples of steeping operations:

EXAMPLE 1

Steeping liquor, containing 17.0% NaOH and 1.5% hemicellulose at 30° C. was applied to the upper surface of a 500 basis weight sheet of cellophane-type pulp (88% alpha cellulose and D. P. 750). The liquor was metered out at a rate calculated to allow about 10 liters of liquor to 1 kilo of pulp and was drawn through the sheet at a rate corresponding to a 20-second time-of-contact with pulp. When the sheet was sucked free of surface liquor, pressure was applied to the alkali cellulose sheet to give a ratio of 2.7:1. The alkali cellulose had an analysis of 33.20% cellulose and 15.36% NaOH, and shredded readily to an open-structured fibrous crumb which was highly reactive and eventually produced a viscose of 8.5% cellulose and 5.25% NaOH composition which had a plugging value of 1520 grams per sq. cm. of filter area.

An alkali cellulose prepared by steeping the same pulp in the same steeping liquor for 30 minutes at 30° C. by a conventional steeping method which is equivalent to single sheet exposure was pressed to the same ratio and gave and alkali cellulose analyzing 33.29% cellulose and 14.12% NaOH. To obtain equivalent alkalinity, the 30 minute sheet-steep required 18.5% NaOH in the steeping liquor which gave 33.32% cellulose and 15.20% NaOH. The resultant viscose filterability from sheet steeping in 17% NaOH was 850 and that from sheet steeping in 18.5% NaOH was 1140. A typical commercial steeping operation under similar conditions except for the use of 26" x 20" sheets in 15-sheet books gave analysis of 33.30% cellulose and 14.52% NaOH (with 18.5% steeping liquor). In this instance the 14.52% is an average including inner spot values as low as 11.00% NaOH, together with outside surface values of as high as 15.60%. The resultant viscose filterability was 605. See the graphs (Figs. 6 to 9) illustrating the relationship of percent NaOH in steeping liquor to percent NaOH in alkali cellulose and to viscose plugging value.

An alkali cellulose prepared by slurrying the same pulp for a 10-minute cycle in 17.0% NaOH at 30° C. followed by pressing to 2.7 gave an alkali cellulose analyzing 32.96% cellulose and 15.20% NaOH. The shredded alkali cellulose consisted of soapy-feeling flakes of a somewhat dense structure which did not disintegrate even with prolonged shredding. The resultant viscose filterability was 690.

EXAMPLE 2

Four 500 basis weight sheets of cellophane-type pulp were steeped as in Example 1. The cycle during which caustic liquor was forced through the sheet was increased to 50 seconds for the four sheets instead of 20 seconds for one sheet. The alkalinity of the pressed alkali cellulose and the resultant viscose filterability were substantially the same for the four sheets as for the single sheet.

EXAMPLE 3

A 450 basis weight sheet of tire cord-type southern pine pulp (94.5% of alpha cellulose D. P. 1150) was steeped as in Example 1. The cycle of forcing caustic liquor through decreased to 10 seconds.

EXAMPLE 4

A 2000 basis weight sheet of cellulose pulp was treated with steeping liquor containing 16.5% NaOH and 3.0% hemicellulose at 40° C. in such manner that the liquor applied to one side of the pulp sheet was drawn through the pulp in an amount corresponding to 5 liters of liquor per 1 kilo of pulp and during an exposure cycle of 40 seconds. The web was sucked to a ratio of 3.5:1 and pressed to a ratio of 2.7:1. The distribution of NaOH in alkali cellulose was uniform and amounted to 14.65% at a cellulose content of 33.70%. When processed into viscose of 8.5% cellulose and 5.25% NaOH content, using 31% CS_2 based on alkali cellulose in xanthation, the plugging value was 1250 grams per sq. cm. of filter area.

Figs. 6 to 9 are graphs illustrating the comparative results of steeping cellulose pulp with caustic soda liquor by conventional methods and by the method of the invention. The captions and values on which the curves are based clearly establish the important practical advantages of the invention.

The following tables illustrate the results of typical operations embodying the invention and comparative results of the method of the invention and conventional cellulose steeping practices. In the tables, pulp A contained 88% alpha cellulose and had a nitrate D. P. (degree of polymerization) of 750, pulp B 94.5% alpha cellulose and a nitrate D. P. of 1150, and pulp C 96.5% alpha cellulose and a nitrate D. P. of 1450. The D. P. was determined by the method described in Analytical Chemistry, vol. 21, pp. 1497–1499 (1949). In the tables, A. C. means alkali cellulose.

Table I

TIME AND OTHER CONDITIONS FOR STEEPING VARIOUS HEMI CONTENT PULPS

Pulp Sheet	Percent NaOH in Steep Liquor	Percent Hemi in Steep Liquor	Force-Through Time in Seconds	Press Ratio [2] after Force-Through
A	17.0	0	19	3.5
		1.5	20	3.6
		3.0	25	3.7
A	17.0	0	17	3.6
		1.5	20	3.6
		3.0	23	3.7
B	17.0	0	9	2.9
		1.5	10	3.0
		3.0	12	3.2
C [1]	17.0	0	4	3.9
		1.5	5	4.0
		3.0	7	4.1

[1] Low density sheet.
[2] This ratio obtained by suction alone.

Table II

EFFECT ON "FORCE-THROUGH" TIME OF SHEET THICKNESS AND PLASTIC LIQUOR TO PULP RATIO

Pulp Sheet	Amount of Steep [1] Liquor Used (ml. liquor/g./ pulp)	Number of Sheets of Pulp	Force-Through Time in Seconds
A	5	1	15
		2	25
		3	37
		4	46
A	10	1	21
		2	32
		3	43
		4	51
A	20	1	25
		2	37
A	40	1	34
		2	45
A [2]	10	1	6

[1] Steep liquor 17.0% NaOH, 1.5% hemi.
[2] Sheet prewet with steeping liquor.

Table III

COMPARATIVE ALKALI CELLULOSE ANALYSIS AND VISCOSE PLUGGING VALUES CORRESPONDING TO VARIOUS METHODS OF STEEPING

Pulp Sheet	Steeping Method	Steeping Liquor		A. C. Analysis [1]		Plugging Value
		Percent NaOH	Percent Hemi	Percent NaOH	Percent Cell.	
A	Sheet	22.0	1.5	17.35	33.40	150
	Slurry	22.0	1.5	18.15	33.05	5
	Force-Through	22.0	1.5	18.57	33.61	25
A	Sheet	20.0	1.5	16.00	33.35	700
	Slurry	20.0	1.5	16.84	32.75	175
	Force-Through	20.0	1.5	17.18	32.85	380
A	Sheet	18.5	1.5	15.20	33.32	1,140
	Slurry	18.5	1.5	16.00	32.88	370
	Force-Through	18.5	1.5	16.25	33.23	950
A	Sheet	17.0	1.5	14.12	33.29	850
	Slurry	17.0	1.5	15.10	32.95	690
	Force-Through	17.0	1.5	15.36	33.20	1,520
A	Sheet	16.0	1.5	13.40	33.14	330
	Slurry	16.0	1.5	14.42	32.56	675
	Force-Through	16.0	1.5	14.67	32.88	1,500
A	Sheet	15.0	1.5	12.80	32.96	100
	Slurry	15.0	1.5	13.73	32.50	450
	Force-Through	15.0	1.5	14.17	33.15	1,110
A	Sheet	14.0	1.5	12.10	32.95	30
	Slurry	14.0	1.5	13.15	32.50	200
	Force-Through	14.0	1.5	13.48	32.95	600
A	Sheet	12.0	1.5	10.80	32.50	5
	Slurry	12.0	1.5	11.90	32.35	25
	Force-Through	12.0	1.5	12.20	32.48	60

"Sheet"=12-sheet book of 10" x 10" sheets steeped 30 min. at 30° C., 20:1 liquor to pulp.
"Slurry"=disperse fibers 10 min., 10 sec. to form each sheet, 30° C., 20:1 liquor to pulp.
"Force-Through"=sheets steeped 21 sec. at 30° C., 10:1 liquor to pulp.
[1] All A. C. pressed to 2.7 press ratio.

Table IV

EFFECT OF LIQUOR EXPOSURE CYCLE ON A. C. ALKALINITY AND ON VISCOSE FILTERABILITY

Pulp Sheet	Steeping Method	Percent NaOH in Steep Liquor Containing 1.5% Hemi	Exposure Cycle	A. C. Analysis, Percent NaOH	Plugging Value
A	Sheet	17.0	20 sec.	11.59	3
	Sheet	18.5	do	12.80	30
	Slurry	17.0	do	14.76	360
	Force-Through	17.0	do	15.36	1,520
A	Sheet	17.0	1 min.	12.50	10
	Sheet	18.5	do	13.55	280
	Slurry	17.0	do	15.06	640
	Force-Through	17.0	do	15.35	1,490
A	Sheet	17.0	2 min.	19.90	80
	Sheet	18.5	do	14.00	450
	Slurry	17.0	do	15.15	660
	Force-Through	17.0	do	15.35	1,570
A	Sheet	17.0	5 min.	13.25	200
	Sheet	18.5	do	14.30	600
	Slurry	17.0	do	15.15	685
	Force-Through	17.0	do	15.35	1,505
A	Sheet	17.0	10 min.	13.50	420
	Sheet	18.5	do	14.55	830
	Slurry	17.0	do	15.15	690
	Force-Through	17.0	do	15.36	1,480
A	Sheet	17.0	30 min.	14.00	790
	Sheet	18.5	do	15.20	1,100
	Slurry	17.0	do	15.15	690
	Force-Through	17.0	do	15.36	1,530

Table V

EFFECT OF AMOUNT OF LIQUOR USED ON A. C. ALKALINITY AND ON VISCOSE FILTERABILITY

Pulp Sheet	Liquor Used Ml./g. Pulp	Cycle (Seconds)	A. C. Analysis, Percent NaOH	Plugging Value
A	5	19	15.26	1,340
A	10	21	15.36	1,520
A	20	27	15.40	1,505

NOTE.—17.0% NaOH, 1.5% hemi in steeping liquor.

Table VI

EFFECT OF TYPE OF PULP AND PERCENT B. D. ON FORCE-THROUGH TIME, A. C. ALKALINITY AND VISCOSE FILTERABILITY

Pulp Sheet	Pulp Density	Percent B. D. of Pulp	Steep Liquor, Percent NaOH	Cycle (Sec.)	Press Ratio	A. C. Analysis, Percent NaOH	Percent CS_2 Used in Xanthation (Percent Based on Cell. in A. C.)	Type Viscose		Plug Value
								Percent Cell.	Percent NaOH	
A	0.80	94	[1] 17.0	21	2.7	15.36	31	8.5	−5.25	1,520
A		50	[1] 17.0	17	2.7	15.40	31	8.5	−5.25	1,190
C	0.73	94	[2] 17.0	8	2.6	15.16	34	7.5	−6.5	3,105
D	0.46	94	[2] 17.0	4	2.6	15.05	34	7.5	−6.5	2,200

[1] 1.5 hemi in steep liquor.
[2] Hemifree steep liquor.

We claim:

In the treatment of dry cellulose with caustic soda steeping liquor to form alkali cellulose followed by removal of excess liquor, the improvement which comprises passing a dry sheet of cellulose pulp of indefinite length continuously through an operation in which the steeping liquor is applied to one side of the sheet and a reduced pressure on the opposite side to impregnate the sheet with the liquor, subjecting the impregnated sheet to mechanical pressure and simultaneously applying to the area of the sheet where the mechanical pressure is applied a reduced pressure to remove by vacuum excess liquor, leaving in the pulp a uniformly distributed amount of sodium hydroxide.

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