

May 3, 1932.

F. H. McCORMICK ET AL

1,857,100

ABSORBENT PAPER

Filed Aug. 25, 1931

4 Sheets-Sheet 1

FIG. 1

- A. % INCREASE IN FREENESS RESULTING FROM TREATING CHEMICAL PULP WITH NaOH SOLUTIONS OF MERCERIZING STRENGTH CORRESPONDING TO TEMPERATURES OF $-5^{\circ}\text{C}.$, $+25^{\circ}\text{C}.$, AND $+100^{\circ}\text{C}.$ FOR SHORT PERIODS OF TIME, (10 MINUTES), AT A 20% CONSISTENCY.
- B. MERCERIZING ACTION OF NaOH SOLUTIONS AT $25^{\circ}\text{C}.$ ON CHEMICAL PULP AS SHOWN BY THE INTENSITY OF THE STAIN PRODUCED BY ZINC CHLORIODINE.

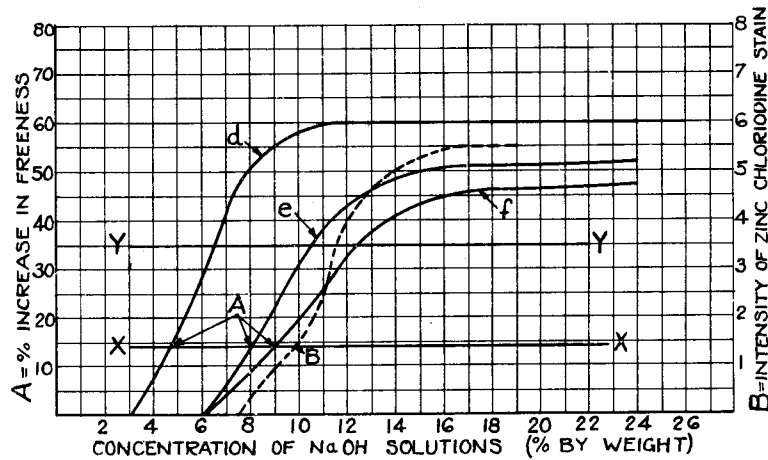
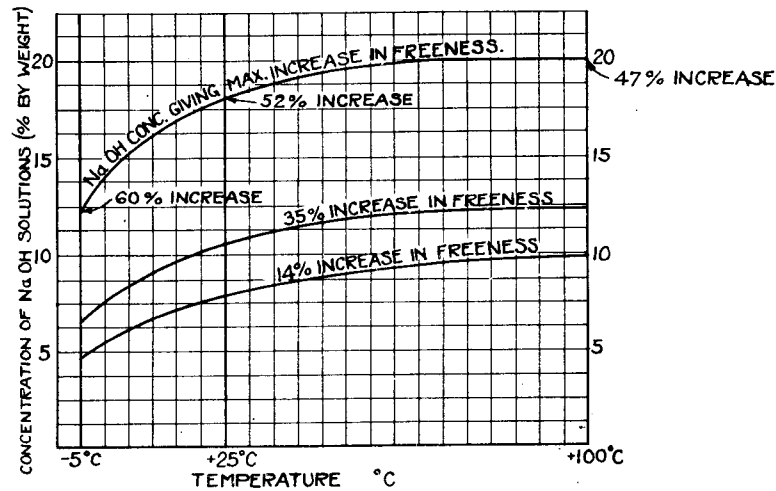


FIG. 2

TEMPERATURE-CONCENTRATION COMBINATIONS THAT PRODUCE DEFINITE PERCENTAGE INCREASES IN FREENESS WHEN CHEMICAL PULP IS TREATED WITH NaOH SOLUTIONS FOR 10 MINUTES AT A 20% CONSISTENCY.



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FIG. 3

EFFECT OF TIME ON THE WEIGHT LOSS OF KRAFT PULP BY TREATMENT WITH NaOH (18% CONCENTRATION 25°C., AND 20% CONSISTENCY.)

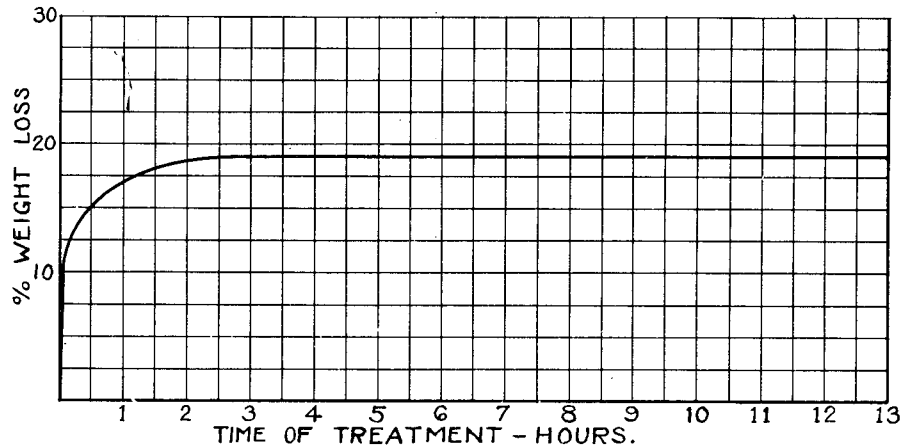
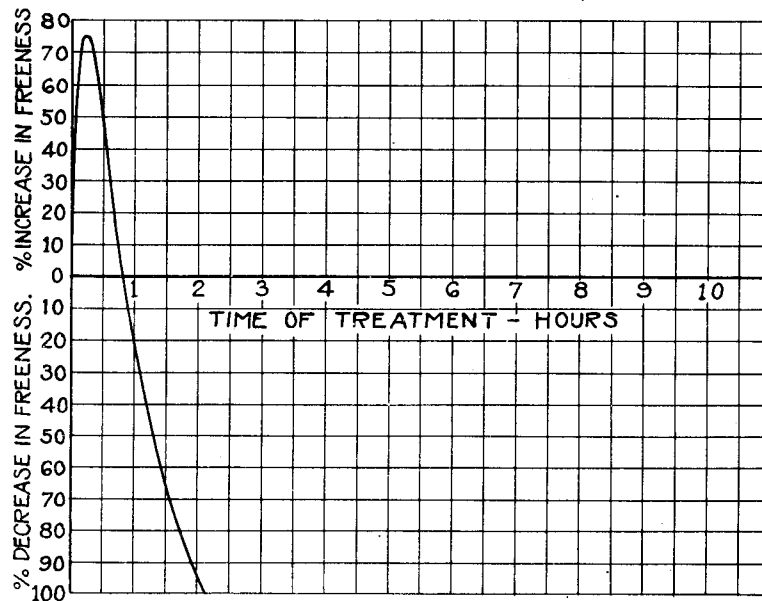


FIG. 4

THE % CHANGE IN FREENESS RESULTING FROM TREATING BLEACHED SULPHITE PULP WITH 18% NaOH (25°C. FOR 5 MIN., 10 MIN., 20 MIN., 30 MIN., AND 2 HOURS, AT A 20% CONSISTENCY.)



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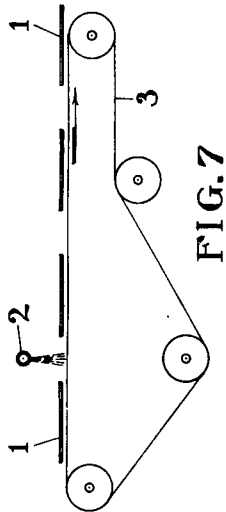


FIG. 7

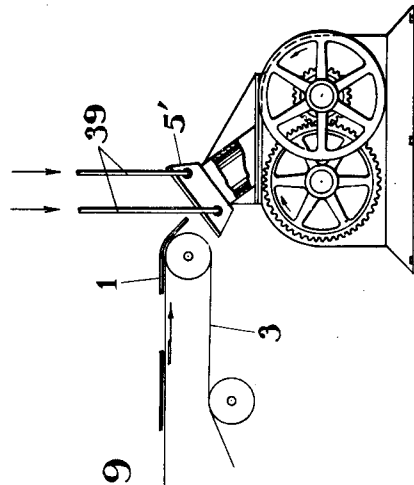


FIG. 9

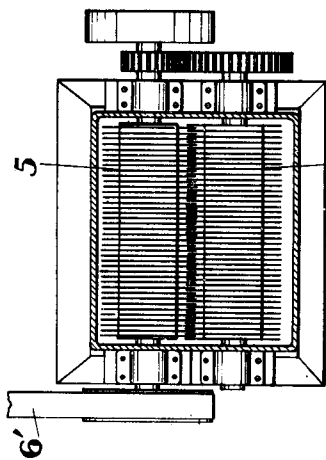


FIG. 5

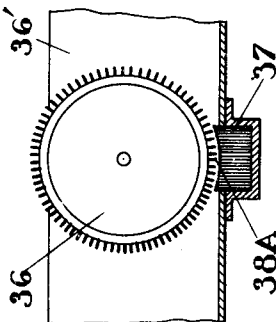


FIG. 6

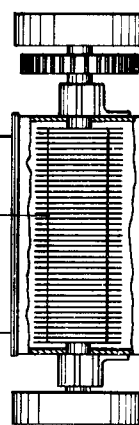


FIG. 8

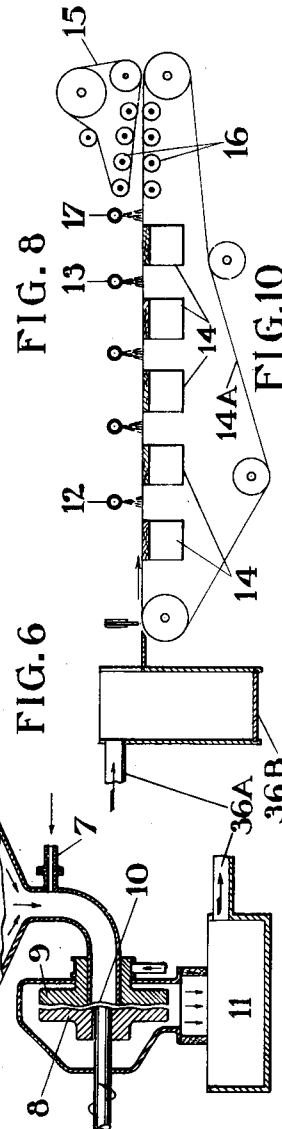


FIG. 10

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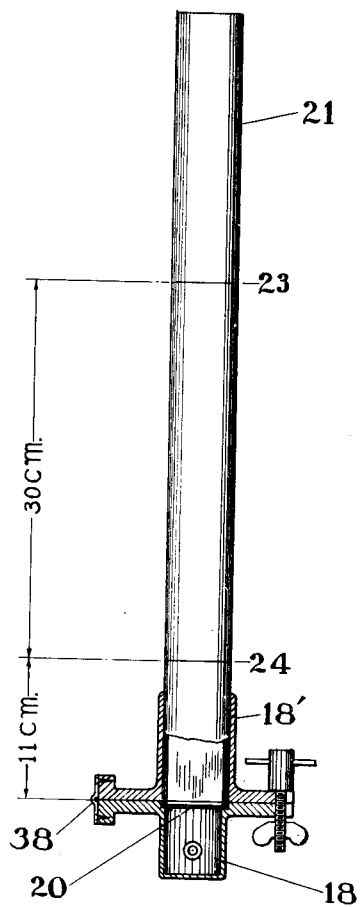


FIG. 11

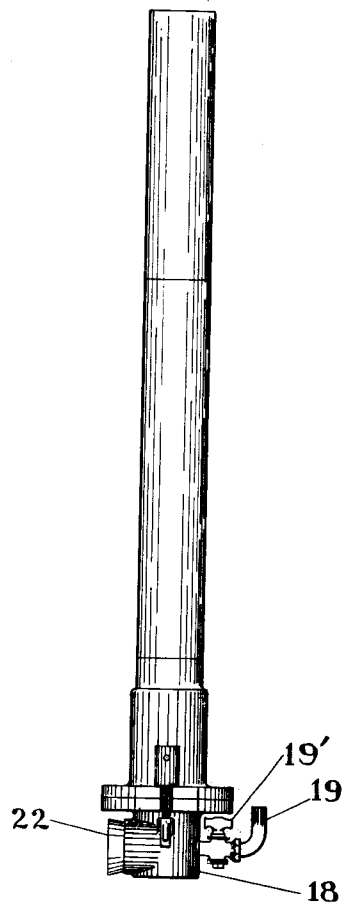


FIG. 12

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UNITED STATES PATENT OFFICE

FRANK H. McCORMICK AND GEORGE L. SCHWARTZ, OF WILMINGTON, DELAWARE, ASSIGNORS, BY MESNE ASSIGNMENTS, TO THE CELASTIC CORPORATION, OF ARLINGTON, NEW JERSEY, A CORPORATION OF DELAWARE

ABSORBENT PAPER

Application filed August 25, 1931. Serial No. 559,302.

This invention relates to a process of producing absorbent paper, and more particularly it relates to the treatment of fibers in pulp form such as fibers from various woods and other materials containing cellulose for the production of materials having new and unusual properties with respect to absorbency, strength, and intensity of curl or crinkle in the fibers. This case is a continuation in part of our application Serial Number 283,445 filed June 6, 1928.

It has been the practice of manufacturers of absorbent papers to obtain good wetting properties of fibers by cooking the pulp in a dilute alkaline solution such as a 2% solution of soda ash, sodium hydroxide, or milk of lime. Rag stock or cotton linters have been used by manufacturers because fibers from these sources respond most readily to the treatment and are not degraded so rapidly as wood fibers. In addition, the linters fibers have a greater natural curl than other cellulose fibers so that the porosity of paper from linters is relatively great.

In the United States patent to Clapp 1,038,086, there is described a method of producing a puffed paper which consists in immersing a sheet of paper in strong caustic and drying the saturated sheet in air. The absorption of carbon dioxide causes the formation of sodium carbonate crystals which produces the puffing effect in the paper. This mode of procedure results in a product having an untreated core with consequent diminution of porosity and inability to permit impregnating media to be uniformly absorbed through the sheet. The long contact of the fibers with the strong caustic and the restricted movement of the fibres when treated in sheet form in the manner disclosed in the mentioned patent, or in the special manner disclosed in Patent 1,757,756 issued to George L. Schwartz, prevents the formation of a product having the high degree of crinkle in the fibre and uniform density and fibre quality throughout as is obtained by our method of intensely crinkling pulp and making the absorbent paper therefrom.

The treatment of cellulose fibres with caustic for the production of pulp and the fur-

ther treatment of the pulp with caustic for various purposes are well known in the fibre art. It has also been proposed to treat fibres in lengths longer than pulp lengths with caustic for the purpose of crinkling the fibres to make them spinnable, or to give them a wool like appearance. Referring more particularly to the treatment of fibres of pulp length and in pulp form, a treatment with which the present invention is concerned, the chief purpose of the prior processes involving the treatment of cellulose fibres in pulp form has been to make products having a high alpha-cellulose content, the treatment involving long caustic digestion to remove hemi-celluloses, such as the treatment of pulp with caustic of mercerizing strength while the pulp is subjected to the mechanical action of a ball or baffle mill for a sufficient time to give a pulp that will make a high grade paper. Another method that has been used for making high alpha-cellulose fibre consists in treating pulp at room temperature with caustic of less than mercerizing activity for the particular pulp used for a period of time sufficient to effect a solution of a substantial proportion of the non-alpha-cellulose content of the pulp. The treatment of cellulose with strong caustic prior to the manufacture of viscose or cellulose derivatives has also been practiced but the discovery that cellulose fibres in pulp form, after being treated with caustic of mercerizing activity for a short period of time as disclosed herein, produce when felted a product having remarkable and valuable properties as in impregnating base has escaped prior investigators.

There has also been described a process consisting of treating rags and paper scraps with caustic of less than mercerizing strength for two hours for the production of soft porous paper containing fibres described as being curled, the product being characterized by the fact that it is capable of receiving impressions even under the slightest pressure. But the present process of treating fibres in pulp form with caustic alkali under the specific conditions of caustic strength and time as hereinafter described for the production of a pulp of extraordinary freeness and a

highly absorbent paper or felt which is characterized by an intense crinkle in the fibres of an entirely different order of magnitude than found in prior products; by high strength in view of its porosity; and by resilience with consequent inability to receive impressions, has not, insofar as we are aware, been disclosed hitherto.

This invention has as an object the production of absorbent pulp or felts from cellulose fibres using a mercerizing reagent under conditions of temperature and concentration and time of treatment that result in the fibres having a permanent and marked curl or crinkle. A further object is an absorbent pulp comprising artificially and intensely curled and kinked fibres from which a highly absorbent felt may be made. A further object is the production of an absorbent felt that is suitable as a base for impregnating compositions such as compositions containing resins, pyroxylin, rubber and the like. Other objects will appear hereinafter.

These objects are accomplished by mixing the pulp with sodium hydroxide solution of mercerizing activity, promptly dispersing the fibres with the solution, promptly removing the sodium hydroxide solution by washing or neutralization, or both, preparing the treated pulp for sheet formation, and then forming sheets on a paper making machine. For the production of our more porous highly absorbent product we use the higher strengths within the mercerizing range and after washing out the caustic directly make the pulp into felt.

We have found that cellulose fibres, whether having some natural curl or not, such as kraft, jute, soda pulp, rope, sulfite and flax can be curled by treating the fibres in pulp form with aqueous solutions of sodium hydroxide of certain concentrations and within certain time and temperature limits. After the treatment is completed the excess caustic solution is removed by pressure or centrifuging and is then washed free with water or neutralized with acid.

In our process there are presumably two distinct reactions of the caustic involved. Of these one may be a solvent action by which the caustic dissolves out oils, resins and other impurities which interfere with wetting. By means of a second reaction, the caustic attacks the cellulose itself. This reaction stops short of actual gelatinization, but does result in a permanent kinking or curling of the fibre. According to our invention we have found that the solvent action proceeds most actively at high temperature and the curling action most actively at low temperature. We have also found that in order to develop both properties to a maximum degree, the fibre may, if desired, be treated first at high temperature and subsequently at a lower temperature.

For a more complete understanding of the present invention reference may be had to the accompanying drawings in which,

Fig. 1 shows by means of curves the increase of freeness obtained when cellulose fibres are treated in pulp form with sodium hydroxide of mercerizing and non-mercerizing activity at a consistency of 20% for 10 minutes;

Fig. 2 is a graph showing the temperature-concentration combinations which produce definite percentage increases in freeness;

Fig. 3 is a graph showing the effect of time on the weight loss which results from the treatment of pulp with sodium hydroxide;

Fig. 4 is a graph showing the percentage of change in freeness resulting from the treatment of chemical pulp with sodium hydroxide for varying periods of time;

Fig. 5 illustrates a form of apparatus that may be used in carrying out the invention;

Fig. 6 is an elevational view partly in section of another mechanism for carrying out the invention;

Fig. 7 is a diagrammatical view of the conveyor for supplying the mechanism of Fig. 8 with pulp;

Fig. 8 is an elevational view of the upper portion of Fig. 6 showing arrangement of the hopper and conveyor;

Fig. 9 is a plan showing the arrangement of the toothed rollers in the mechanism of Fig. 8;

Fig. 10 shows the conveyor on which the treated pulp is washed or washed and felted;

Figs. 11 and 12 are elevational views of an apparatus for determining the freeness of the treated pulp.

In the treatment disclosed herein, the fibres, caustic strength, temperature and time of treatment are important factors, and these should conform to the following specification:

The fibres must be in pulp form during the caustic treatment, and by pulp it is to be understood that we mean cellulosic fibrous material consisting of fibres not arranged into sheets and short enough and sufficiently low in diameter to be made into a uniform felt from a water suspension. The fibres must be of paper-making length, and long fibres, such as those separated from the raw materials by some form of degumming process, will not give the kind of product with which the present invention is concerned. Fibres that may be designated as paper making length have a minimum mean length of about $\frac{3}{4}$ m. m. and a maximum mean length of about 4 m. m. Because of the fact that fibres having a mean length of less than $1\frac{1}{2}$ m. m., such as the fibres of non-coniferous wood pulp, do not crinkle as intensely as the longer fibres within the paper-making range, we prefer, especially when working with the weaker solutions within the mercerizing range to use

fibres having a mean length of $1\frac{1}{2}$ to 4 m. m. Non-coniferous pulps of long fibre length, such as rope, are shortened by cutting and/or beating to the proper fibre length. The strength of the caustic solution must be within the range known as mercerizing activity. The caustic content of a solution that may be designated as being of mercerizing activity at any given temperature is well understood by those skilled in the fibre art. A caustic solution of this strength improves the luster of fabrics and also improves the dyeing properties and tensile strength. The dye absorbency of the fibres of paper pulp is also increased, the X-ray pattern changed, the ease of hydration reduced, and the fibre changed from a flat ribbon form to a rod-like form. It is to be understood, however, that the present invention is not concerned with mercerization as such, the term mercerization being used herein simply as a convenient means for expressing the activity of the caustic used in the present process. The concentration of caustic required at a particular temperature to bring the activity of the solution to the proper value within the mercerizing range is also dependent somewhat upon the kind of fibre used. Thus a 15% solution of sodium hydroxide at 25° C. is required to give rope fibres the same curl that is obtained in kraft fibres with a 12% solution at the same temperature. Non-coniferous pulps, especially those produced by the sulfite process, also require a higher caustic concentration in the solution as will be seen from the fact that a 10% sodium hydroxide solution at 25° C. is below mercerizing strength for this pulp, whereas mercerizing action begins on kraft pulp with an 8% caustic solution at the same temperature. In general, the range of operative concentration of the present invention may be stated as from 8% sodium hydroxide and up since solutions above this concentration are within the operative range at room temperatures for the majority of the pulps that we prefer to use. The operative concentration range at temperatures lower than room temperature will be lower as will be apparent from the fact that a 4.7% concentration at -5° C. is equivalent to an 8% concentration at room temperature.

While the process may be operated between the freezing point of the caustic solution (-10° C. for sodium hydroxide) and +104° C., it is ordinarily not desirable to operate at the lower and the higher temperatures because the gelatinizing action at the lower temperatures and the degrading action at the higher temperatures takes place with such rapidity that it is difficult to effect distribution of the caustic throughout the pulp and to wash out the caustic to stop the reaction in the extremely short time required to prevent the mentioned gelatinizing and degrading action

from decreasing the amount of crinkle in the fibres below the desired value. It is preferred, therefore, to operate at temperatures not far removed from room temperatures. In general, high temperatures cause the fibres to become wet more quickly with the caustic solutions and thus react more readily without preliminary treatment, but very high temperatures, especially in the presence of small amounts of air, such as are absorbed from the atmosphere during treatment, tend to degrade the fibres. However, by controlling the time of treatment at high temperatures the degrading action can be held within practical limits. Also high temperatures tend to reduce the degree of curling. According to our invention the best conditions for producing good wetting properties and a good curl are 20° C. to 40° C. with an 18% aqueous solution of sodium hydroxide. Lower temperatures tend to curl the fibres more than high temperatures and some fibres tend to gelatinize on the surfaces if temperatures below 10° C. are used. Temperatures below 20° C. also tend to reduce the wetting properties of the treated fibres.

The actual time of contact between the fibres and the caustic solution of mercerizing activity is much shorter than in the prior processes involving the treatment of pulp with caustic solutions. The present invention is based upon the discovery that the treatment of fibres in pulp form with strong caustic for a short period of time causes the fibres to become intensely crinkled and that the product obtained by felting these fibers is extremely porous and has a remarkably high impregnated strength. The same result cannot be obtained by treating pulp with solutions of mercerizing activity for long periods of time, or by treating the pulp with a solution below mercerizing activity for long or short periods of time. In practicing the present invention the reaction between the pulp and the strong caustic must be stopped before a solution of most of the non-alpha cellulose content of the pulp takes place, the maximum time permissible being usually less than thirty minutes with high pulp consistencies. The optimum theoretical conditions for operating the present process would be to stop the reaction by washing out the caustic the instant after it has wetted the pulp. This is of course not easily accomplished in practice because the mechanical distribution of the solution throughout the pulp, and separation of the caustic from the pulp requires an appreciable time. The effect of time on freeness is illustrated in Fig. 4 wherein the maximum freeness at a 20% consistency is attained in about 10 minutes. It must be noted, however, that this time includes the time required to disperse the fibers by the caustic and mechanical means. The crinkling is substantially simultaneous with the wetting

of the fiber with the mercerizing solution and if a high ratio of liquor were used to facilitate quick wetting the peak of the freeness curve in Fig. 4 would be much closer to the zero ordinate. It will be seen from Fig. 4 that after the maximum increase in crinkle, (which is measured by the increase in freeness as hereinafter explained), is attained, the quality of the product obtained rapidly depreciates with time until the crinkle in the fiber is less than the initial value.

As noted above a short time of contact between the fibers and the caustic solution of mercerizing activity is essential in the practice of our invention, but when speaking of the actual time of contact it is necessary to take the consistency, i. e., the ratio of pulp to liquor, into consideration, because when using a low ratio of sodium hydroxide to fiber a larger proportion of the sodium hydroxide content of the solution is used up or inactivated by the initial mercerizing action so that the effective activity of the reagent is reduced. This has the effect of a reduction in the time of treatment. When using high consistencies it will be apparent then that while the mechanical operation of dispersing the pulp into the caustic solution and washing out the caustic may be extended longer than would be permissible with a high ratio of liquor, the actual time of contact of the fibers with a solution of the mercerizing activity initially used is in fact very short. It will be seen therefore that the use of a high consistency is a convenient means for insuring sufficient time for completion of the necessary mechanical manipulations while at the same time preventing extended time action of the strong caustic on the fibers. A sufficient volume of liquor is necessary to permit dispersion of the fibres but within this limit we prefer a high consistency. An excess of caustic beyond that required for easy wetting of the fibres is undesirable because of the greater weight loss, greater degrading, and the lower residual curling of the fibers remaining after the necessary mechanical steps in the process have been completed. Very high ratios of liquid to pulp make it difficult, when working on a large scale, to complete the steps of wetting the pulp and stopping the reaction in time to prevent loss of the soluble non-alpha cellulosic constituents and consequent diminution of the crinkling of the fibers below the required value. The use of a large volume of liquor also makes the practice of the process less economical because of the cost of recovering the caustic which involves evaporation of the large quantity of water that must be added to dilute the solution below mercerizing activity to stop the reaction between the pulp and caustic. The mechanical difficulties offered by dilution with the required large volume of water are also considerable when low consistencies are

used. Low consistencies offer less difficulties when working on a small scale, as for instance stirring the fibers into the solution in a small vessel with a paddle and diluting with water immediately after the fibers are wet with the solution. In this case the best results would be obtained in less than a minute and a time as long as twenty minutes would be undesirable. In the practice of our invention we prefer to use ratios of solution to pulp such as will give pulp consistencies from 15% to 25% since we have found that the use of these consistencies overcome the difficulties encountered when it is attempted to carry out the process with the low pulp consistencies (consistencies low enough to be pumped from one vessel to another and seldom higher than 5% to 6%), which are conventionally used in processes for treating pulp.

The prompt dispersion of the pulp into free fibers by the mercerizing agent and mechanical treatment is essential to give the fibers freedom to crinkle and to insure treatment of each fiber with a solution of sufficient concentration to produce the desired degree of crinkle. Especially in high consistencies the selective absorption of sodium hydroxide from water by cellulose fibers is so great that treatment of pulp without prompt dispersion gives a spotty effect in which only part of the fibers are crinkled. If the dispersion is accomplished without abrasive action none of the softened cuticle of the fiber is torn off and the weight loss is held at a minimum. The softening of the fiber cuticle will be held at a minimum and loss of valuable material prevented if the reagent is removed promptly as soon as the surfaces of all fibers are made wet with it. A satisfactory way for separating the solution from the fibers to stop the reaction is to wash with water or a dilute solution of the reagent which will reduce the concentration below mercerizing activity. Further removal of the agent may be accomplished more slowly if necessary without increasing unduly the weight loss or without lowering the degree of crinkle.

Results obtained by treating the pulp with sodium hydroxide of the required activity and within the temperature and time limits mentioned will be more clearly understood by reference to the curves shown in Fig. 1, the curves A, designated *d*, *e* and *f*, showing the percent increase in freeness of kraft pulp obtained by treating it at a 20% consistency for 10 minutes at -5° C., $+25^{\circ}$ C., and $+100^{\circ}$ C., respectively, with sodium hydroxide solutions of varying concentrations. The freeness of the pulp is measured by the time required for the water to drain from an aqueous dispersion of the pulp and this is also a measure of the degree of crinkle in the fiber. The method used for measuring the freeness of ordinary pulps cannot be used

with any degree of accuracy because of the extreme freeness of our treated pulp, a special apparatus being designed for this purpose as will be more fully described in connection with Figs. 11 and 12.

The ordinates of the curve B are the various intensities of the stain produced by zinc chloriodine on kraft pulp which has been treated with caustic of various concentrations within the mercerizing range at 25° C. For most pulps, as kraft and sulfite pulps, with the exception of those of non-coniferous origin, sodium hydroxide of 8% concentration at 25° C. is of mercerizing activity as indicated by the intensity of the zinc chloriodine stain. It will be seen from curve (e) that, when operating at normal temperature, an 8% caustic treatment within the short time permitted by the present process results in a 14% increase in the freeness of the pulp, and that the percent increase in freeness becomes rapidly greater as the caustic concentration rises to about 18%. Above this concentration a very slight additional increase in freeness takes place until the concentration reaches about 24%, above which it is not desirable to go because the slight additional increase in crinkle does not justify the expense of higher caustic concentrations, and the wetting properties of the solution gets poorer, requiring at a 35% concentration a too large excess of the strong solution for complete wetting. At temperatures lower than 25° C. lower concentrations are required to give solutions of the same reactivity or strength. Thus for temperatures between -10° C. and -5° C. the solution is of mercerizing activity at about 5% concentration which also causes a 14% increase in freeness as indicated by the curve d which is drawn to show the increase in freeness with increasing caustic concentrations at -5° C. Likewise higher concentrations of caustic are required for higher temperatures, a concentration of above 9% being required to bring the solution within the mercerizing range as indicated by curve f at 100° C. and to give the same increase of freeness as is caused by an 8% concentration at 25° C.

We also point out that fibres react under these conditions according to their characteristics and the preliminary treatment that they have received. Thus, fibres from poplar contain a great deal of material soluble in caustic solutions under these conditions and a considerable loss in weight occurs, while in cotton there is sometimes a slight gain in weight. This gain in weight is probably due to chemical combination of water and cellulose. However, if the cotton has been severely degraded by cooking or heat-aging there is a loss in weight, and other properties are changed. If uncooked cotton is used the temperature is preferably high to facilitate wetting. If kapok fibres are used the tem-

perature may likewise be high to insure wetting, or a small amount of wetting agent, such as a sulfonated oil, may be added. It will be understood, therefore, that for different fibres the concentrations may have to be raised or lowered to produce the desired increase in freeness since variations of 2% are, in some instances, required to produce the same effect on different pulps as previously pointed out in connection with rope fibres and non-coniferous pulps. For the purpose of the present invention we therefore define the minimum activity of the solution as one which will, within the operable temperature and time limits of the invention, cause the freeness of the pulp to increase more than 14%. The line $x-x$ corresponds in ordinate height to the increase in freeness caused by solutions just above minimum mercerizing strength, and the intersection of this line with the various temperature curves gives the concentration required at the particular temperature to bring the solution just above minimum mercerizing strength. Similarly, higher strengths within the mercerizing range are so defined and we have drawn the line $y-y$ to show the caustic strength required at different temperatures to cause a 35% increase in freeness when the time of treatment is too short to cause excessive removal of non-alpha cellulose constituents.

Now our invention resides in the discovery of the new products which result from treating fibres of paper-making length in pulp form with mercerizing solutions for short periods of time which do not cause excessive removal of the non-alpha cellulose constituents, and more specifically our invention resides in the discovery that when the treatment is with a solution above the activity required to increase the freeness more than 35% and up to 60% (requiring above 10.5% caustic concentration at 25° C. for kraft pulp, above 5% concentration at the lower temperature limits, and above 12.5% caustic concentration at the higher temperature limits as indicated by the intersection of line $y-y$ with the curves in Fig. 1), the intensely crinkled fibres of the pulp yields a paper or felt capable of absorbing unusually large quantities of colloidal dispersions, or other viscous impregnating media while retaining a high degree of pliability.

When the short crinkling treatment is with caustic having a strength not greater than required to give the 35% increase in freeness, but above mercerizing activity (between 8% and 11% concentration at 25° C.), the crinkled pulp, while it does not produce by direct felting a product having the exceptional absorbent properties mentioned above, does produce when beaten in water a hydrated product which felts into a denser but somewhat less porous product which has an impregnated strength comparable to leather.

The caustic concentration at any particular temperature that is required to give the crinkling effect mentioned above may be expressed in terms of the concentration of a solution at a definite temperature that possesses the requisite activity to cause an increase in freeness of more than 14%. We have therefore, for convenience, defined the concentration required to give the necessary mercerizing activity as one having a mercerizing activity equivalent to that of an aqueous sodium hydroxide solution of 8% to 35% concentration at 25° C. For the production of our highly crinkled pulp that has been increased more than 35% in freeness the solution used must have a mercerizing activity equivalent to a sodium hydroxide solution having a concentration between 10.5% and 35% at 25° C. The amount of caustic that a solution must contain at any given temperature within the operative range to give a solution having a mercerizing activity equivalent to that of a solution containing 8 to 35% sodium hydroxide at 25° C. may be easily determined by those skilled in the art in accordance with the principles discussed in connection with the curves in Fig. 1. Thus in making the highly crinkled pulp having a freeness increase greater than 35%, any concentration from 6.5% to 9% may be used within the range of -10° C. to +15° C. Concentrations of 10.5% and above may be used at any temperature between 15° C. and 100° C. It is most practical, however, to operate with concentrations from 10.5% to 24% at temperatures within the range of 15° C. to 40° C. and an 18% concentration at room temperature is preferred. When preparing the lesser crinkled pulp, which has a freeness increase of 14% to 35%, and which is capable of being hydrated by long beating in water for the production of the product referred to above, we usually use in practice any concentration between 8% and 11% with a temperature within the range of 0° C. to 100° C.

The dry fibres may be added to an 18% solution, in sufficient amount to wet the pulp entirely, or the fibres may be added in wet form if the concentration of the solution is sufficiently higher to compensate for the water in the pulp. The mass should be agitated long enough (a few minutes with efficient mixing apparatus) to insure complete wetting, and excess caustic should then be removed by pressing or centrifuging. This pulp is then put into a washer where it is freed from caustic solution or the last traces of caustic may be neutralized with acid. If kraft, sulfite or mechanical wood pulp are used, the fibres are usually short enough to make into paper without further beating. If linters or rope or other long fibres are used, they will require some treatment in the beaters or Jordan, or both, before they can be

made into paper. The pulps from these fibres are then made into porous paper using either a Fourdrinier or a cylinder machine. Various modifications of the standard machine may be used to conform to the peculiar "freeness" of the pulp but these vary with the kind of fibre used or the proportion of treated to untreated fibres used.

An apparatus which may be used in carrying out the invention and which illustrates the required type of treatment for the distribution of the caustic throughout the pulp is the ordinary beater, referred to above and shown in Fig. 5, which consists of a receptacle 36' in which the power driven roll 36 may be raised so that the longitudinal bars 38A may clear the bed plate 37. The caustic solution and air-dry pulp in a ratio to give a consistency of 15% to 25% (from 5.7 parts to 3 parts of solution to one part of pulp) is put into the beater and the roll raised slightly from the bed plate as shown in the drawings so that the bars 38A will squeeze the pulp against the bed plate and separate the individual fibres from the pulp mass and secure contact between the fibres and the caustic solution and cause wetting of the fibres in the shortest possible time without any abrasive or cutting action. Air-dry pulp, or pulp exposed to normal atmosphere, contains about 8 to 12% moisture. A standard trade practice is to designate air-dry pulp as 90% bone dry or containing 10% moisture. The caustic concentrations are adjusted to compensate for the known amount of moisture in the pulp. The consistencies mentioned herein are upon the conventional basis of the ratio of bone dry weight of fibre to liquid. As soon as the mixing is sufficiently complete to insure wetting of substantially all the fibres a stream of water is run into the mixer to stop the reaction and the beater roll is run until the fibres are washed to neutralization. The reaction between the caustic and the fibres ceases immediately with the dilution of the caustic by the water. Since the desired reaction between the fibres and caustic is practically instantaneous and since beating or drastic treatment of the fibres during the mixing is undesirable it will be apparent that the best mode of practicing the invention involves mixing the solution with the pulp in an apparatus which squeezes the pulp without abrasive action and disperses it into the solution, and then diluting or washing out the caustic alkali to stop the reaction after a time not appreciably longer than required to completely disperse the fibres throughout the solution.

Unlike the usual treatment of pulp with caustic for the production of high alpha cellulose, the beta and gamma cellulose and other non-alpha cellulosic constituents of the fibres are not regarded as impurities in the

practice of our process. Comparing Figs. 3 and 4 it will be seen that with increase in time the freeness decreases as the weight loss increases. It is desirable, therefore, to operate under conditions which do not result in substantial solution of the non-alpha cellulose constituents. The yield in the practice of our invention is generally 90% to 95% and with specific arrangements for speeding up the dispersion of the fibres into the solution, the yield may approach 98%.

The freeness of the pulp is a measure of the curling or crinkling in the fibres which has taken place as a result of the treatment herein disclosed. Pulp treated by the present process, however, allows the water of the dispersion thereof to drain through it too fast for accurate measuring by the usual methods of determining pulp freeness. We have therefore used the apparatus disclosed in Figs. 11 and 12 in measuring absolute freeness values. It will be understood of course that the percent increase in freeness is independent of the method of measuring. The numeral 21 designates a glass tube $1\frac{1}{8}$ inches inside diameter having a metal cup 18 hinged at 38 to a base 18' on the tube which holds a wire screen 20 of 0.006 inches thickness and 50 x 70 mesh over the bottom of the tube. The cup 18 and base 18' have cooperating lugs opposite the hinge slotted to receive the threaded shank of a fastener extending from a head which rests on the lug extending from 18'. The cup 18 is filled with water until it flows out of the pipe 19 which fixes the water level in the cup even with the top of the screen 20 so that the fibres of the pulp dispersed in the tube will not mat against the screen. The stop cock 19' is then closed and a dispersion of the pulp (5 g. bone dry weight in enough water to make 1000 cc. at 25° C.) is then poured into the glass tube 21. The stopper 22, which closes an orifice $1\frac{1}{2}$ inches in diameter is promptly removed and as the level of the water of the pulp dispersion passes the 41 centimeter mark designated by the numeral 23, the stop watch is started. As it passes the 11 centimeter mark indicated by the numeral 24 the watch is stopped. The time elapsing in seconds for the water to drain through 30 cm. between the two points 23 and 24 is the freeness.

In the following examples the process has been carried out with the apparatus described in connection with Fig. 5. The sodium hydroxide is a commercial grade containing not more than 2% sodium carbonate.

Example I

	Pounds
Kraft from southern pine dry weight	75
Caustic soda 19.5% aqueous solution at 55° C.	555
Consistency 12%.	

The kraft pulp in sheets containing 60% water was added to the solution in the beater

of 150 pounds capacity, and the roll was held about 3 mm. above the bed plate while the pulp was mixed with the solution. When the solution was distributed throughout the pulp, which required about 15 minutes, a stream of water was run into the beater to stop the reaction, the washing screen was lowered into the pulp and the beater was run until the pulp was practically free from caustic. This required three hours continuous beating and washing. The freeness of the treated pulp as determined by the described method was 27 seconds.

The washed fibers were then pumped into a mixing chest and thence through a Jordan where they were cut very slightly to shorten them in order to make the pulp run through the piping to the paper machine, and were then run into a molding chest where the fibres were formed into a sheet on a cylinder. The sheet of fibers was removed from the cylinder by a wet felt and carried through press rolls to remove excess water and thence over drying cans until dry.

Example II

	Pounds
Kraft from northern spruce	800
Caustic solution 22.2% at 60° C.	3600
Consistency 18.2%.	

The kraft pulp containing 50% dry weight of fibres was added to the solution and the mass was well mixed. The beater roll was held a quarter-inch above the bed plate, water was added after 20 minutes to stop the reaction, the washing screen was let down in the pulp and washing was continued until the pulp was free from caustic. Three hours continuous washing was required to remove practically all of the caustic. The freeness of the pulp was 30 seconds. The fibres were then let down into a storage chest, pumped through the Jordan, without cutting, into the mixing chest, thence pumped to the screens and thence onto the Fourdrinier. From the Fourdrinier the sheet followed a wet felt between press rolls and thence over drying cans until dry. Another part was run over the wet felt without pressure and thence over the drying cans until dry and thence between calender rolls. This latter part was more porous and more pliable than the part that was pressed when wet. The calender treatment when dry was given to increase the pliability.

Example III

	Pounds
Old rope cut into three inch lengths had been cooked under pressure with 1% soda ash and 1% lime	700
Caustic solution 18% at 60° C.	4400
Consistency 13.7%	

The beater roll was held a half-inch above the bed plate and the rope strands went

through the slot readily. In 30 minutes all strands were absent and the larger fibres were considerably dispersed into the smaller fibers. The roll was then lowered to $\frac{1}{8}$ inch from the bed plate. Water was then turned into the beater to stop the reaction and the washer was let down into the pulp. The pulp was practically free from caustic in three hours after the rope was first added to the solution. The freeness of the pulp was 38 seconds. The roll was then let down to a brushing position and beating was continued one hour longer. The rope stock was then ready for the Jordan. Half of this was run through the Jordan with very slight cutting and a very porous absorbent paper was obtained.

Example IV

	Pounds
Sulfitc from Sweden—unbleached and containing some bark fibres called shives	800
Solution caustic soda 20.4% at room temperature	5900
Consistency 11.8%.	

The sulfitc pulp contained 50% water. It was added to the caustic solution in the beater and when completely wet with the solution water was added to stop the reaction, and the screen was lowered into the pulp. The beater roll was a quarter-inch above the bed plate throughout the washing period. After $3\frac{1}{2}$ hours washing the pulp was practically free from caustic and it was run over the Fourdrinier machine, producing a porous absorbent sheet of good strength. This pulp had a freeness of 32 seconds.

As previously pointed out, our process is most economically and satisfactorily carried out by using high pulp consistencies and for these reasons it is usually desirable not to use much more solution than that given in Examples I and IV wherein about 7.4 parts by weight of solution are used for 1 part by weight of pulp.

Similar runs were made with bleached sulfitc and soda pulp. The loss of weight with soda pulp was found to be high, making the use of this material less desirable for many purposes.

Bleached linters were also run on an experimental cylinder machine of 18 inch width giving a paper more porous, absorbent and of greater strength than was obtained from untreated linters.

We have also made runs from a mixture of kraft and sulfitc pulp, kraft and linters, kraft and soda pulp and kraft and rope. Certain advantages are obtained by using mixed fibers for certain uses. For example, the use of treated kraft with treated rope gives a more bulky and more absorbent paper than treated rope alone with the added strength of the rope fibres. We have found that linters when mixed with kraft furnishes a fibre more re-

active to colloiding solutions such as cold caustic solutions of low concentration. Likewise, bleached sulfitc with kraft gives a fibre more reactive to colloiding caustic alkali solutions than kraft alone.

We have also prepared papers from various mixtures of the treated fibers with untreated fibers in order to obtain various degrees of absorbency.

Another form of apparatus for carrying out our process, which in some respects is better adapted for commercial practice, is shown in Figs. 6 to 10, inclusive. Air-dry pulp 1 compressed into the usual commercial pulp board is fed onto a wire screen conveyor 3 where a shower of sodium hydroxide solution of 18% concentration at room temperature falls upon them. The excess caustic solution which runs off the sheet is stored for further use by means not shown. Considerable of the caustic solution is absorbed by the sheets which causes them to swell and soften. These sheets promptly drop from the conveyor into the hopper 5' (Fig. 8) of a shredder shown in plan Fig. 9 and in elevation in Fig. 8. As the sheets enter the shredder they are showered with more caustic solution of 18% concentration from spray pipes 39. The rolls 5 and 6 are armed with teeth which overlap between the rollers and which tear sheets into pieces of about one-half inch in diameter. These rolls are driven by the belt 6' at different speeds, such as 100 R. P. M., and 600 R. P. M., respectively, by means of the smaller gear on the driven shaft of the roll 5 which meshes with the larger gear on the shaft of the roll 6. The pulp then drops down in a disc refiner where additional caustic is supplied through pipe 7 in such quantity as will bring the total amount up to a ratio of 4 parts solution to one part of pulp (20% consistency). As previously noted, the concentration of the solution may be made sufficiently higher than the final concentration desired in order to compensate for the water in the pulp. The pulp then passes to the center of the disc refiner constituted by the discs 8 and 9 where the fibres are dispersed from the mass and wet with the caustic without abrasive action on the fibres. The discs 8 and 9 have smooth interfitting corrugated faces as shown and are driven in opposite directions by a mechanism not illustrated at a relatively high speed and are set with a clearance of $\frac{1}{16}$ inch to $\frac{1}{8}$ of an inch so that each fibre is freed from the mass in the passage 10 which extends from the entrance at the center of the disc to the edges where they are thrown off by centrifugal force. From this disc refiner the pulp drops into a trough 11 where the reaction between the caustic and fibres is stopped by diluting the caustic which also lowers the stock density to a pumping consistency. The duration of the treatment, that is the time

elapsing between the application of the caustic on the conveyor from pipe 2 and the deposition of the pulp into the diluted solution in the tank 11, is not more than 15 seconds. The pulp passes through the conduit 36A to the chamber 36B from whence it passes over a wire screen 14A where it is formed into a thick sheet and washed free from excess caustic solution. While fresh water may be introduced into the trough 11 to wash out the caustic to stop the reaction, we prefer to use the caustic (below 6%) resulting from the counter-current washing system on the screen 14A so as to avoid dilution of the solution to the point where recovery of the caustic from the solution is no longer profitable. The numeral 14 designates a series of suction boxes between which the washing showers of water are directed. The shower 13 is water and the weak caustic washed through the sheet into the last suction box is pumped to the preceding shower forming a stronger solution in the suction box below. The concentration progressively increases until the solution in the second suction box resulting from the shower 12 contains about 5% caustic concentration which is pumped back into the tank 11. The solution from the first suction box, which precedes the shower 12 and which removes the excess weak caustic resulting from the treatment of the pulp in tank 11, is run into a concentrator for recovering the caustic by raising the concentration of the solution to the value required for conducting the process. After passing over the suction boxes the sheet passes under another wire screen 15 where it is pressed between two or more sets of squeeze rolls 16. If the treated pulp is not free from caustic solution before passing through the wires 14A and 15 a shower of diluted hydrochloric acid from the pipe 17 is sprayed onto the sheet ahead of the squeeze rolls. The squeezed pulp is now formed into wet laps which are ready to be charged into the beater for making into paper.

After the pulp is dispersed in the strong caustic solution and is diluted with water or dilute caustic solution, it should be washed free from caustic as soon as possible. Mechanical treatment in this stage is unnecessary but it is not so injurious as in the strong caustic stage. After the pulp has been freed from caustic solution it may be stored indefinitely as wet laps or in dilute pulp form or as dry pulp, and is ready for forming into sheets of felt at any time.

The paper-making stage of the process is carried on in regular paper making equipment with certain modifications indicated below. The consistency of the pulp in the beater is from 3% to 5%. For the preparation of the very absorbent material, only enough beating is done to disperse the fibres.

The pulp is then dropped into chests where it is diluted with more water and it is then sent over a Fourdrinier machine at a consistency of 0.6% to 0.3%. Long agitation in the chests must be avoided to prevent formation of agglomerates and we therefore use a slow shake with an amplitude of at least three eighths of an inch. The Fourdrinier wire has an up-hill pitch of at least one inch for six feet of length so the water can form a pool and so it will not rush over the wire fast enough to disturb the thin sheet of fibers that is formed as soon as the pulp reaches the wire. The pulp is so free that it does not hold water long enough to obtain a satisfactory sheet formation when the wire runs with the down-hill or horizontal pitch. We have also found it desirable to direct a shower of water on the sheet where the water is leaving the surface and draining through the sheet. The sheet then passes under a dandy roll over suction boxes, or suction roll, or both and then through the wet press. Metal top press rolls must be covered with felt to prevent the sheet from sticking. Rubber covered press rolls or stone press rolls require no felt covering. The sheet after leaving the wet press passes over regular paper machine dry cans. When it emerges from the drying system it is run between calender rolls to soften the texture and is then made into rolls and cut with regular paper winding and slitting equipment. Sheets 0.100" thick and above are cut into the desired lengths after leaving the wet presses and are dried in conveyor ovens without canvas felts. Sheets of maximum softness and bulk, especially those 0.100" or above are made by omitting the wet press operation and are dried in conveyor ovens with subsequent pressing between calender rolls.

The process for washing the pulp described in connection with Fig. 10 may be combined with the process for forming the finished sheet. Thus the mixture of pulp and strong caustic in the tank 11 may be diluted with the weak caustic solution of the countercurrent washing system to a consistency of 0.3 to 1.0% and the Fourdrinier machine equipped with a shake so that a well formed sheet is made before it is washed free from caustic. Finally, after the acid shower, a fresh water shower is applied and the wet sheet is run on to regular paper drying equipment without going through the beating process.

When using the equipment described in Fig. 5 and high pulp consistencies such as disclosed in Examples I to IV which are used with this type of apparatus, the best crinkling is obtained if the total elapsed time from the addition of caustic to the pulp, dispersion of the pulp into distinct fibres, and washing the caustic from the fibres, (or diluting the caustic solution below mercerizing activity);

is less than 10 minutes. In plant practice, however, the total time for the apparatus of Fig. 5 usually averages about 15 minutes, but when using the equipment illustrated in Figs. 6, 7, 8, 9 and 10 the total time, which averages only about 15 seconds, is sufficiently short to prevent appreciable diminution in the initial crinkle imparted to the fiber even though low pulp consistencies are used.

Although the foregoing specific embodiments describe the use of an 18% solution of sodium hydroxide for a few minutes, the treatment may be continued considerably longer at room temperature, especially at high consistencies, with certain resistant pulps such as rope, without degrading the cellulose to an appreciable extent.

In the following example the process was carried out as described in connection with the mechanism shown in Figs. 6 to 10, inclusive.

Example V

	Pounds
Kraft pulp, 90% bone dry (freeness on special tester 45 seconds)-----	1, 000
Sodium hydroxide (aqueous solution 20% at 21° C.)-----	4, 000

The pulp was passed through the system with a fixed rate of feeding the solution and pulp so as to give a uniform consistency. The strength of the solution in contact with the fibres during the treatment was 19.5% and the time of treatment with this solution was 15 seconds. The freeness after completing the treatment was 30 seconds. The treated pulp gave a dark blue color with zinc chloriodine reagent.

A sheet of felt was formed from this pulp by the following process: The pulp was dispersed in Hollander beaters with the rolls in light brushing position for a half hour to disperse the fibres. The pulp was then let down into a preparation chest and after the pulp was diluted with some water and stirred just enough to keep it in suspension it was pumped into a Jordan in which the cone was backed off the bars to prevent cutting. Following dilution with more water in a machine chest the pulp was pumped to a flow box above plate screens where it was diluted to a consistency of 0.4%, screened and passed onto a Fourdrinier provided with three slices and having an up-hill pitch of one inch for six feet of length. The shake was thirty per minute at an amplitude of one half inch. The first press roll was felt covered. After drying on regular paper-machine driers the sheet was passed between two calender rolls to soften. The final sheet was .048-.050 inches thick and weighed 0.68 pounds for an area of 36 x 40 inches.

When a 3" square of this sheet is dropped into a 33% natural rubber latex it sinks promptly, the latex coming through the sheet just as rapidly as around the edges. The

latex swells the sheet but it can be conveyed between press rolls with or without screens where it is compressed to about 25% bone dry fibre weight and it is then dried over a regular paper can drier system. If a similar sheet is dropped onto a surface of 15% pyroxilin-castor oil dispersion (five seconds pyroxilin) in organic solvents, it sinks into the dispersion rapidly and is strong enough to pass between doctor blades for removing excess dispersion. The dry unimpregnated sheet has a porosity of 0.13 seconds and a dry impregnated sheet containing 50% rubber from latex based on total weight of pressed sheet has a porosity of 4.0 seconds as measured on a Gurley densometer. Its density is 0.253 which is obtained by dividing its weight in grams by its volume in cubic centimeters. The pliability of a sheet containing 50% rubber from latex based on total weight is 9.0 to 17.5 units on the Pfund pliability tester. It can be bent around a mandrel three fourths of an inch in diameter in either direction without exhibiting a paper break and the impregnated product containing 50% rubber from latex based on the total weight can be bent around a mandrel seven sixteenths of an inch in diameter without exhibiting a paper break. Its Elmendorf tearing strength is 80 to 120 grams and the impregnated product containing 50% rubber from latex based on the total weight has a tearing strength of 1200 to 1800 grams. These values are given for a sheet that contains 0.68 pounds of air-dried fibres per 36" x 40" area with a thickness of 0.045 to 0.050 inches.

Although unbleached kraft fibres are given as the preferred raw fibrous material, it has been found that bleached and unbleached fibres from the following sources have special uses and are amenable to this treatment. These are sulfite, soda pulp, rope (Musa textiles), flax, hemp (Cannabis), rag, and caraoa.

The shredder and disc refiner for dispersing the pulp in the strong caustic solutions that is described in the drawings is used because this type of equipment is commercially available although modifications of the shredder, such as the use of finer teeth and closer spacings, can be employed. Also the disc refiner can be substituted by one or more additional shredders. Or, if pulp as wet lap or from a screw press (40-55% bone dry) is used the conveyor and shredder are not essential and the pulp is passed directly into the feed inlet of the disc pulper where all of the caustic is added at 7 in Fig. 6. A stronger solution is used to allow for extra water in this pulp. In general, any mechanical equipment can be used that will effect prompt saturation of the pulp with strong caustic solution, prompt dispersion of the pulp into individual fibres, and prompt removal of strong caustic solution from this

dispersion. Equipment that permits continuous treatment in contrast to batch treatment is preferable.

For formation into sheets varying from .005" to .060" a Fourdrinier type machine is best adapted. A heavy shower of water in small drops may be applied to the sheet at the area where the free water is just dispersing from the surface in order to smooth out the surface of the sheet. However, a cylinder type machine is satisfactory for certain types of sheets. For very thin sheets a Harper Fourdrinier machine may be used. For sheets above .100" a board machine is preferable.

Sodium hydroxide is the preferred crinkling agent because of its commercial availability, but other caustic alkalies as potassium and lithium hydroxides may be used in concentrations equivalent to the required amount of sodium hydroxide as may be determined by simple chemical computation of mole weights. The reference to sodium hydroxide in the claims is intended to include equivalent strengths of these other caustic alkalies.

Our invention is not limited to the treatment of cellulose fibre with caustic soda solutions. It is well known that there are various chemical reagents which at suitable concentration and temperature are capable of dissolving or dispersing cellulose. In the case of caustic soda it is known that the activity of the reagent increases at low temperatures and is a maximum for certain intermediate concentrations, which in general are within the range known as mercerizing strength, that is, the concentration commonly used in mercerizing cotton goods. As examples of such chemical reagents, we mention the fixed caustic alkalies, sulfuric acid, zinc chloride, calcium thiocyanate, ferric chloride, and cuprammonium solutions. In some cases, for example the caustic alkalies, the activity of the reagent increases with decreasing temperature. In other cases (zinc chloride) the activity increases with increasing temperature. As to the concentration which gives the most active reagent, there seems to be no general rule, but in the case of each of these reagents the concentration and temperature for maximum dispersing activity are known to those skilled in the art of the cellulose industry.

In addition to functioning as solvents or dispersing agents, each of these reagents is capable of acting as a swelling agent when the conditions of temperature and concentration are so chosen that the activity of the reagent approaches but does not actually reach the maximum. When treated by the reagent within these limits the cellulose fibre is swelled and more or less superficially gelatinized but is not dissolved.

Our invention relates to the use of that class of reagents which are capable of acting as cellulose swelling or imbibing agents but we

use these reagents under conditions of temperature time and concentration such that the mercerizing activity of the reagent is substantially equivalent to that of a sodium hydroxide solution between 8% and 35% concentration at 25° C. and approaches but does not reach the stage required to bring about gelatinization of the cellulose. We have discovered that when used under these conditions the reagent induces a reaction with the cellulose which stops short of gelatinization but which causes the cellulose fibre to take on a pronounced and permanent kink or curl.

In the case of zinc chloride, it was found that a 70% solution at 40° C. curls and kinks kraft fibres and unbleached linters. Bleached linters are gelled too much under these conditions, but at 70° C. unbleached and bleached linters both dissolve and kraft is slightly gelled. At a temperature and concentration of 40° C. and 70% the time required is less than two minutes. In commercial practice we prefer, however, to use a 65% solution at 65° C. for a ten minute period because of the difficulties in getting thorough mixing if the solution and fibre in less time. The solution may contain between 50% and 68% zinc chloride at 65° C.

For ferric chloride hexahydrate, we prefer to employ a solution consisting of 80% hydrate and 20% water at 50° C. to 100° C. Solutions containing as low as 50% of the hydrate are effective at 100° C.

All of the following examples are given for a ten minute period of treatment. It is desirable, however, to use shorter time if the mechanical conditions for plant operation will permit.

When using sulfuric acid we prefer a 60% concentration at 20° C. Concentrations between 40% and 65% are satisfactory but a concentration of 65% is slightly too reactive at 20° C.

When using cuprammonium the optimum temperature is room temperature and the most satisfactory concentrations range between 0.4% and 0.5% Cu in the solution. The operative concentration range at room temperature is from 0.3% Cu and 0.7% Cu.

Solutions of calcium thiocyanate are satisfactory when the solution contains between 40% and 55% of $\text{Ca}(\text{CNS})_2$ at 100° C. It is preferred to use solutions containing 55% $\text{Ca}(\text{CNS})_2$ at 100° C. Solutions of higher concentration and temperature gelatinize the fibres.

When using the reagents mentioned below the general procedure which was used for treating the fibers follows: The chemical wood pulp was treated for 10 minutes at 5% consistency with the crinkling solution at a certain temperature. This treatment was carried out by mixing 5 parts by weight of air dry pulp and 95 parts by weight of the solution in an open container, with sufficient

stirring to give thorough dispersion and good wetting of all the fibers. The treated fibers were then washed with water until free of the cellulose swelling reagent. The change in appearance of the fibers effected by the treatment was determined by microscopic examination. The preferred time for treating the fibers with the solution before washing with water depends on (1) the efficiency of the method used for mixing the fibers and solution, and (2) the time required for the solution to swell the fibers, which is a matter of seconds. With very efficient mixing apparatus, a time of one minute or less would be ample. It is undesirable, even with very low ratios of liquor to pulp, to continue the treatment longer than 20 to 30 minutes. Also, with efficient mixing apparatus the consistency at which the pulp is treated is preferably around 15% to 20%; that is, 15 parts to 20 parts of air dry pulp treated with 85 parts to 80 parts of solution.

Calcium chloride-zinc chloride: Highly curled and kinked fibers were prepared by treating kraft pulp with a water solution containing 52% by weight of calcium chloride (anhydrous) and 10% by weight of zinc chloride (anhydrous) at 135° C. The pulp has a freeness of 80.5 seconds before treatment and of 33.5 seconds after treatment.

Reagents which are moderately less reactive than sodium hydroxide are included in the following:

Hydrochloric acid: Moderately curled and kinked fibers were prepared by treating kraft pulp with hydrochloric acid (37% by weight of hydrogen chloride) at 25° C. The pulp had a freeness of 80.5 seconds before treatment and of 48 seconds after treatment.

Sheets were formed by standard procedure from the treated pulp. A sheet weighing 0.68 pounds per 36" x 40" area had an Elmendorf tearing strength of 160 grams, and a sheet of the same weight impregnated with rubber latex to give 39% by weight of rubber in the dried sheet had an Elmendorf tearing strength of 960 grams. The sheets impregnated with rubber were pliable and free from paper break.

Sodium metasilicate: Moderately curled and kinked fibers were prepared by treating kraft pulp with a 37% solution of sodium metasilicate at 25° C.

Still other reagents that may be mentioned as effective for curling and kinking chemical wood pulps are aluminum chloride at 37% concentration and 100° C., glacial acetic acid at 25° C., phosphoric acid at 85% concentration and 25° C., barium bromide at 58.3% concentration and 100° C., and potassium formate at 85% concentration and 100° C.

While we prefer to conduct the pulp treating process as described above, the process may also be carried out by other methods in which the fibers are not too restricted me-

chanically from movement. We desire it to be understood, therefore, that the treatment of the fibers in pulp form as mentioned in the claims includes also the treatment of a mat or sheet of pulp provided the individual fibers have sufficient freedom of movement and are not compacted or felted as in a finished sheet of paper. For example, a mat may be formed on a Fourdrinier or cylinder machine from untreated fibers from a water suspension or from a caustic soda dispersion below mercerizing activity and while the mat is still in a loose uncompact condition, before heat drying, the caustic soda solution of mercerizing activity may be applied to the surface of the sheet. The force of gravity will carry the mercerizing solution through the sheet and all of the fibers will be crinkled "in situ," though not to the same degree as when they are dispersed in the form of pulp. Or when still further crinkling is required, the fibers treated in pulp form may be dried as loose pulp, or as loose sheets, and then redispersed to form sheets, thus adding still more bulk to the finished sheet.

The pulp treated in accordance with the present invention is characterized by its unusually high freeness as compared with that of the normal untreated pulp. When the treatment is with solutions of the lower activity required to give a freeness increase of at least 14%, the absolute freeness value is seldom slower than 65 seconds and for the highly crinkled pulp, the absolute freeness value serves to identify the pulp and for many pulps is as fast as 15 seconds. The pulp having a freeness increase of more than 35%, the absolute values usually ranging from 40 seconds for pulp having a freeness increase of 35% to 15 seconds for pulp having a freeness increase of 60%, is directly felted into the highly absorbent products presently described, and the lesser crinkled pulp having freeness values corresponding to 14% and 35% increase in freeness is especially useful for making the hydrated product previously referred to.

The treated pulp which we prefer to use and which is the most valuable for making our absorbent felt has an absolute freeness of from 15 seconds to 25 seconds, has been increased in freeness 52% or more, and is obtainable by using sodium hydroxide solutions between 17.5% and 35% concentration, at room temperature.

When the process has been carried out by sodium hydroxide the treated pulp resulting from the short caustic treatment followed by washing, may be identified, in addition to its freeness, by the zinc chloriodine test which distinguishes pulps or papers treated with sodium hydroxide solutions of mercerizing activity. While pulps treated with this strength of caustic for periods longer than those which characterize the present inven-

tion also respond to the zinc chloriodine test, they are differentiated by their freeness since they seldom have a freeness as determined by the method described herein faster than 60 seconds. Pulps that have been manufactured felt will be better appreciated by the comparison in the following table with papers made by treating pulp with caustic outside of the conditions of time and concentration disclosed herein. In the table

10 Sheets	Porosity Seconds		Tearing strength Grams		Paper break Cylinder size		Pliability		75
	Unimpreg- nated	Impregnated	Unimpreg- nated	Impregnated	Unimpreg- nated	Impregnated	Unimpreg- nated	Impregnated	
A.....	(1) 0.13-0.26 (2) 0.26-0.35	3.6-6.6 6.6-50	80-150 150-175	1000-1800 1000-2100	1 1/16"-3/4" 3/4"-1"	3/16"-3/4" 1/4"-3/8"	3.8-7.2	7.7-12.0	
15 B.....	1.2-2.3	13-20	150-200	900-1200	1 1/4"-2"	1 1/2"-7/8"	15-24	18-29	80
C.....	1.3-3.2	13-22	150-200	900-1400	1 3/4"-2 1/2"	3/4"-1"	26-40	30-44	
D.....	1.8-3.3	66-200	100-150	200-400	1 1/2"-3"	1/2"-1"	27-40	28-42	
E.....	0.47	20.8	264	975	7/8"	3/16"	14.8	11.5	
20 F.....	0.35	22.0	79	380	1 1/16"	1 1/16"	5.5	10.3	85
G.....	0.17-0.37	24.4-75.6	142-412	2000-2075	1 1/16"-7/8"	3/4"-3/4"	3.1-12.5	3.5-6.6	

by the soda process in which wood chips are cooked with 8% to 11% of sodium hydroxide at high temperature and pressure show only a slight coloration easily distinguished from that obtained when the pulp is further treated with caustic as described herein.

The following table gives a comparison of the increase in freeness resulting from the practice of the invention as compared to the increase in freeness obtained by other pulp treating processes:

Pulp	Freeness in seconds	Treatment	Freeness after treatment in seconds
40 Kraft....	45	18% NaOH at 25° C. for 10 min. at 20% consistency.	21.2
Kraft....	45	11% NaOH at 25° C. for 10 min. at 20% consistency.	26.9
Kraft....	45	NaOH just above mercerizing activity (8% or slightly above at 25° C.), at 20% consistency.	38.0
45 Kraft....	45	6.1% NaOH at 100° C. for 2 hours at 5% consistency.	46.1
Kraft....	45	6.1% NaOH at 100° C. with beating for 2 hours at 5% consistency.	84.6
A commercial absorbent pulp containing above 94% alpha cellulose.....			75.0
Blotter pulp.....			327.0

It is to be understood that the freeness values in the claims refer to the values obtained when the fibers are dispersed in the stated amount of water and the test carried out in accordance with the specifications hereinbefore given. It is to be noted that a high freeness is expressed in a small number of seconds and that a low freeness is expressed in a large number of seconds.

Our highly crinkled pulp may also be identified by making it into paper and applying certain tests to the sheeted pulp. The valuable properties with respect to porosity, tearing strength, paper break, and pliability for the unimpregnated and impregnated paper or

The letter A designates a felt made from kraft pulp treated in accordance with the present invention at a consistency of 20% for 15 minutes. The values in the horizontal line with the numeral (1) indicates the properties of our preferred product made from pulp treated with 17.5% to 35% sodium hydroxide at 25° C. (or with solutions of equivalent activity), and the values in the horizontal line with the numeral (2) indicate the properties of our felt made from pulp treated at room temperature with 10.5% to 17.5% sodium hydroxide or its equivalent.

The letter B designates a paper made from a pulp treated with 18% sodium hydroxide at 25° C. at a consistency of 6% for 30 minutes in a ball mill. The data here shows the effect of abrasive action in dispersing the pulp into fibers.

The letter C designates a paper made from kraft pulp treated at a consistency of 6% with sodium hydroxide just below mercerizing activity, (between 7.5% and 8%) for 30 minutes.

The letter D designates a known paper that has been made by treating pulp at a consistency of 5% with sodium hydroxide solution of 9° Bé. (6.1%) and heating for two hours at 100° C.

The letter E designates an absorbent paper made from cotton linters.

The letter F designates an absorbent paper known as roofing felt which contains a mixture of cellulose and wool fibers and is usually made from rags.

The letter G designates a commercial absorbent paper consisting of very high (above 94%) alpha cellulose.

The values in the above table are given for a sheet containing .68 pounds air-dry fibers per 36" x 40" area with a thickness of 0.045" to 0.050".

All samples were impregnated with 50%

rubber latex excepting B, C and D which were saturated but were incapable of absorbing enough to give a product containing 50% rubber.

The porosity values were obtained with the Gurley densometer, a standard machine in the paper industry. It measures the rate at which 100 cc. of air is passed through a circular disc of the material. The circular orifice has an area of 1 sq. inch. The pressure is obtained from a 20 oz. cylinder that fits into another cylinder. The lubricant is a low viscosity mineral oil. For the very porous products the readings are made on a 300 cc. air displacement but are calculated to 100 cc. for purposes of comparison. The figures for the tearing strength were obtained with the Elmendorf tear tester which is a standard testing equipment in the paper industry. The results are expressed as the sum total force in grams required to tear 16 individual sheets, eight with the machine direction and eight against. The sheets are $2\frac{1}{2}$ " square and are notched $\frac{1}{2}$ " with a knife at the point where the tear begins. The standard conditions are 50% relative humidity at 25° C.

Comparisons as to resistance to paper break are made by stating the size of the smallest cylinder or mandrel around which the sheet may be rolled without the appearance of paper break. The pliability units were obtained by means of a special apparatus known as the Pfund pliability tester which is not described in detail inasmuch as comparative values only need to be considered. Latex was selected as the impregnating medium because the results are easily duplicated and because the results are typical of the effect of impregnating material in general.

It is to be noted that the residual impregnated porosity of 50 seconds for product (2) under A in the table includes papers made from pulp other than kraft which have a much lower initial freeness before treatment than sulfate pulp. When kraft pulp is used the residual impregnated porosity would not be more than 12 seconds for the product resulting from the 10.5% sodium hydroxide treatment.

It is to be understood that the paper sheet referred to in the claims in connection with the mentioned physical characteristics is defined in terms of these units and that it contains .68 pounds air-dry fiber per 36" x 40" area with a thickness of 0.045" to 0.050".

In addition to the physical characteristics previously listed our new product is characterized by the substantially circular cross sectional form of the fibers, which results from the treatment with mercerizing solutions as herein disclosed, as distinguished from the ribbon-like form of chemical wood pulp

fibers that have not been treated with solutions of mercerizing activity.

When the pulp has been crinkled with sodium hydroxide the zinc chloriodine test may be applied as a further aid in identifying the paper.

The zinc chloriodine solution for carrying out the test should be prepared fresh for each test and may be made by adding 4 drops of a solution of 1 gram of iodine and 20 grams potassium iodide in 100 cc. of distilled water to 20 cc. of a solution of 280 grams zinc chloride dissolved in enough distilled water to make 300 cc. About 0.50 grams air-dry pulp is dispersed in a test tube with 100 cc. distilled water, the excess water drained off in a Gooch crucible and expressed by pressure to about 75% moisture. The pulp is transferred to a test tube containing 5 cc. of zinc chloriodine solution at a temperature of about 20° C. to 25° C. and shaken until dispersed. The prompt occurrence of blue color indicates that the pulp has been treated with about 8% to 35% sodium hydroxide. If the color is very deep it indicates that the concentration of the solution (at room temperature) was above 13% and graduations of lighter shades indicate concentrations down to the point where mercerizing activity begins. Mixtures of treated and untreated fibers are recognized under the microscope after staining with zinc chloriodine solution. The color persists several days with bleached sulfite pulps treated by the present process. Unbleached kraft pulps treated likewise give a color almost black due to the brown color of the pulp and it fades within a few hours. This test is also used for the felt made from the pulp, impregnating and coating materials being separated from the fibers by suitable solvents.

Our highly crinkled pulp when felted into sheet form exhibits unusual porosity toward colloid dispersions and there is no surface filtering effect during impregnation as is exhibited by blotting paper. Gurley densometer tests show that this unusual porosity to colloids is due to the large pores uniformly distributed throughout the sheet. Microscopic examination shows that these pores are due to the crinkled condition of the fibers and to the unusual properties that the treated fibres have in precipitating the impregnating material onto their surfaces which prevents filling the interstices. These materials when impregnated with colloids exhibit unusual strength and they are more pliable after impregnation to a given strength than other saturated papers. The porosity of some of the impregnated products, especially those impregnated with rubber latex, is sufficient to absorb glues readily so that the edges may be glued like leather. The Gurley densometer measurements show that this porosity is equivalent to that of ordinary split leather. The

scope of raw materials from which these products are made is very great and this is a decided advantage in stabilizing the cost and quality of the products. The products absorb glues more readily than ordinary saturating papers. They also have unusual absorption and retention properties for hydrophilic colloids. Sheets of these products are more uniform than sheets from ordinary saturating papers. The impregnated products can be skived as readily as leather. Where the products are impregnated with fusible impregnating media, unusually large quantities of these media can be held without migration resulting from gravity or heat changes.

The remarkable properties of our product which makes it especially valuable as an impregnating base will be apparent from the comparison of the porosity, tearing strength, paper break, and pliability values given in the appended table. We wish to call attention to the high porosity values for our product and particularly to the high porosity value which is obtained after the sheet is impregnated with 50% latex. This residual porosity after impregnation is highly important when it is desired to add a finishing coating for various effects in the manufacture of artificial leather or where breathing properties are required such as in inner soles. The unimpregnated tearing strength is high in view of the extreme porosity of the product and the impregnated strength is higher than most other papers similarly impregnated. With respect to paper break it will be seen that the maximum size of the cylinder around which paper break occurs for both the unimpregnated and impregnated product is smaller than in the case of the other samples. The pliability values for the impregnated and unimpregnated sheet also indicate a pliability much higher than is usually found in impregnated products.

The pulp produced in accordance with the present invention may be formed into a highly absorbent felt which has been found to be a valuable base for impregnation with various impregnating media such as resins, rubber, etc.

The unimpregnated products may be used as filter media for air, solids from liquids, heat insulation, padding and sound-proofing. The unimpregnated base may also be used as raw material for vulcanized fibre. The impregnated products are used as a base for artificial leather, for gasket materials, belting, shoe parts, electrical insulation, floor coverings, and wall coverings.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that we do not limit ourselves to the specific embodiments

thereof except as defined in the following claims:

We claim:

1. A process of producing absorbent pulp comprising dispersing cellulose fibers of paper making length in pulp form with a swelling agent and stopping the reaction between the pulp and swelling agent when the crinkling of the fibers is sufficient to produce a freeness increase above 14%.

2. A process of producing absorbent pulp comprising dispersing cellulose fibers of paper making length in pulp form with a swelling agent and stopping the reaction between the pulp and swelling agent when the crinkling of the fibers is sufficient to produce a freeness increase of 35% or more in the freeness of the pulp.

3. A process of producing absorbent pulp which comprises dispersing cellulose fibers of paper making length in pulp form with a mercerizing agent having a mercerizing activity equivalent to that of an aqueous sodium hydroxide solution between 8% and 35% concentration at 25° C., and separating said agent from the fibers before solution of a substantial proportion of the non-alpha cellulose content of the fibers takes place.

4. A process of producing absorbent pulp which comprises dispersing cellulose fibers having a mean length of $1\frac{1}{2}$ to 4 mm. in pulp form with a caustic alkali solution of mercerizing activity, and diluting the solution below mercerizing activity to stop the reaction before the percent freeness increase of the fibers passes below 14%.

5. A process of producing absorbent pulp which comprises dispersing cellulose fibers of paper making length with a caustic alkali solution of mercerizing activity at a consistency of 15% to 25%, and diluting the solution below mercerizing activity in less than 30 minutes to stop the reaction.

6. A process of producing absorbent pulp which comprises dispersing cellulose fibers of paper making length in pulp form with caustic alkali solution having a mercerizing activity equivalent to that of an aqueous sodium hydroxide solution between 10.5% and 35% concentration at 25° C. and diluting the solution below mercerizing activity before the percent freeness increase of the fibers passes below 35%.

7. A process of producing absorbent pulp which comprises dispersing cellulose fibers of paper making length in pulp form with a solution of mercerizing activity in an apparatus which disperses the pulp into the solution without abrasive action on the fibers, separating the solution from the fibers after a time not appreciably greater than required to completely disperse the fibers with the solution, and washing the solution from the fibers.

8. A process of producing absorbent pulp

which comprises dispersing cellulose chemical paper pulp with an aqueous solution of sodium hydroxide above 10% concentration at a temperature of 20° C. to 40° C., and promptly
5 diluting the solution below mercerizing activity.

9. The method of producing absorbent pulp comprising mixing cellulosic fibrous pulp material with caustic alkali solution of
10 sufficient strength such as will produce a crinkling or curling of the fibers and in such volume as will be sufficient to wet the fibers but insufficient to give a ratio of solution substantially greater than about 7.4 parts
15 by weight of solution for one part by weight of pulp, and washing the pulp.

10. The method of producing absorbent pulp according to claim 9 including recovering excess caustic solution from the pulp
20 mass of crinkled fibers and forming it into sheets of highly absorbent material.

11. A step in the method of producing absorbent pulp by curling the fibers thereof, this step consisting in impregnating pulp with
25 caustic alkali solution of 8 to 35% concentration and maintaining the fluid content of the mass of pulp and solution in such amount as will not be appreciably greater than required to wet the pulp and cause dispersion of the
30 pulp with the caustic.

12. The method of producing absorbent pulp comprising delivering cellulosic fibrous pulp to a mixing apparatus, delivering caustic
35 alkali to said mixing apparatus to produce in the apparatus a curling or crinkling of the fibers and to produce a ratio of solution to pulp such as will not be appreciably greater than required to wet the pulp and cause good dispersion of the pulp with the caustic, delivering
40 the pulp so treated to a tank, delivering weaker caustic alkali to said tank, delivering the pulp to mechanism for removing excess liquor, delivering the pulp to a washer, and delivering the washed pulp to a paper
45 making machine and forming it into sheets of highly absorbent material.

13. The method set forth in claim 12 in which the caustic alkali is supplied in such
50 quantity as to give a volume of liquor sufficient to wet the fibers but insufficient to give a ratio of solution substantially greater than about 7.4 parts by weight of solution for one part by weight of pulp, and adjusting the
55 concentration of the caustic alkali as to compensate for the water content of the pulp supplied to the mixing apparatus.

14. The method set forth in claim 12 in which caustic alkali solution is recovered
60 from the pulp prior to washing the pulp.

15. A process of producing absorbent paper which comprises dispersing cellulose paper pulp with a solution of mercerizing activity, stopping the reaction between the solution
65 and fibers before the per cent freeness increase of the fibers passes below 14%, washing

the solution from the fibers and making the fibers into paper.

16. A process of making absorbent paper which comprises dispersing cellulose paper pulp with a solution having a mercerizing
70 activity equivalent to that of an aqueous sodium hydroxide solution between 10.5% and 35% concentration at 25° C., diluting the solution below mercerizing activity to stop the reaction before the per cent freeness increase of the fibers passes below 35%, washing
75 out the solution and making the fibers into paper.

17. A process of making absorbent paper which comprises dispersing cellulose fibers of
80 paper making length with a solution of mercerizing activity, diluting the solution below mercerizing activity to stop the reaction before the per cent freeness increase of the fibers passes below 14%, washing out the solution and
85 felting the fibers into paper on a Fourdrinier wire having an up-hill pitch.

18. A process which comprises dispersing cellulose fibers of paper making length with
90 a solution of mercerizing activity, diluting the solution below mercerizing activity to stop the reaction before the per cent freeness increase of the fibers passes below 14%, washing out the solution, beating the fibers only enough to disperse them, diluting to a consistency of 0.3% to 0.6%, agitating just enough
95 to avoid formation of agglomerates, felting the fibers into paper on a Fourdrinier wire having an up-hill pitch of at least 1 inch for 6 feet of length, directing a shower of water on the surface of the sheet where the water is
100 leaving the surface, passing the sheet through a wet press, and drying the sheet.

19. Paper pulp comprising artificially crinkled superficially mercerized cellulose fibers
105 having a freeness of 15 seconds to 40 seconds, said fibers having the substantially circular form in cross section which characterizes cellulose fibers treated with solutions of mercerizing activity.

20. Paper pulp comprising artificially crinkled superficially mercerized cellulose fibers
110 which has a freeness of 15 seconds to 40 seconds and which exhibits a blue stain when treated with zinc chloriodine solution.

21. Paper pulp comprising artificially crinkled superficially mercerized cellulose
115 fibers which has a freeness of 15 seconds to 28 seconds and which exhibits a blue stain when treated with zinc chloriodine solution.

22. A highly absorbent paper comprising
120 felted artificially crinkled cellulose fibers having a freeness of 15 to 28 seconds, said fibers having the substantially circular form in cross section which characterizes cellulose fibers treated with solutions of mercerizing activity.

23. An absorbent paper comprising felted
125 artificially crinkled cellulose fibers having a freeness of 15 seconds to 40 seconds, and a po-

rosity of 0.13 seconds to 0.35 seconds, said fibers having the substantially circular form in cross section which characterizes cellulose fibers treated with solutions of mercerizing activity.

24. An absorbent paper comprising felted artificially crinkled superficially mercerized cellulose fibers having a freeness of 15 seconds to 28 seconds, and a porosity of 0.13 seconds to 0.26 seconds, said fibers exhibiting a blue stain when treated with zinc chloriodine solution.

25. An absorbent paper comprising felted artificially crinkled cellulose fibers having a freeness of 15 seconds to 40 seconds, said paper being characterized by its capability of being impregnated with 50% rubber latex while retaining sufficient pliability to be folded around a cylinder of from $\frac{1}{8}$ to $\frac{3}{8}$ inch in diameter without paper break, said paper when so impregnated being also characterized by a residual porosity of 3.6 to 50 seconds, and a tearing strength above 1000 grams, said fibers having the substantially circular form in cross section which characterizes cellulose fibers treated with solutions of mercerizing activity.

26. The process of manufacturing absorbent material from cellulose fibers, which comprises curling said fibers by treating with a swelling agent a mixture of reactive and less reactive cellulose fibers in pulp form, washing out the swelling agent, and making the fibers into felt.

27. The process of manufacturing absorbent material from cellulose fibers, which comprises curling said fibers by treating them in pulp form with a swelling agent of mercerizing activity, promptly washing out the swelling agent, mixing therewith untreated fibers, and making the fibers into felt.

28. The process of manufacturing absorbent material from cellulose fibers, which comprises curling said fibers by separately treating with a swelling agent two grades of the fibers in pulp form, one less reactive to the swelling agent than the other, washing out the swelling agent from each, mixing the two grades of fibers, and making the fibers into felt.

29. The process of manufacturing absorbent material from cellulose fibers which comprises curling said fibers by treating them in pulp form with an aqueous caustic solution above normal room temperature, lowering the temperature to room temperature, washing out the caustic and making the fibers into felt.

30. A puffed highly absorbent paper which comprises a sheet containing a mixture of artificially curled and uncurled cellulose fibers, said curled fibers having a freeness of 15 to 40 seconds and having the substantially circular form in cross section which charac-

terizes cellulose fibers treated with solutions of mercerizing activity.

31. A puffed highly absorbent paper which comprises a mixture containing 25% by weight of artificially curled cellulose fibers and approximately 75% of untreated cellulose fibers, said curled fibers having a freeness of 15 to 40 seconds and having the substantially circular form in cross section which characterizes cellulose fibers treated with solutions of mercerizing activity.

32. A process of producing absorbent pulp which comprises dispersing cellulose fibers of paper making length in pulp form with a solution of mercerizing activity in an apparatus which disperses the pulp into the solution without abrasive action on the fibers, separating the solution from the fibers after a time not appreciably greater than required to completely disperse the fibers with the solution, washing the solution from the fibers, and felting the treated fibers into absorbent sheet material.

33. A process of producing absorbent pulp which comprises dispersing kraft paper pulp with an aqueous solution of sodium hydroxide above 10% concentration at a temperature of 20° C. to 40° C., promptly diluting the solution below mercerizing activity, and felting the treated fibers into absorbent sheet material.

34. A process for making absorbent material which comprises treating cellulose fibers of paper making length in uncompacted condition permitting freedom of movement of the individual fibers with a mercerizing agent having a mercerizing activity equivalent to that of an aqueous sodium hydroxide solution of mercerizing activity between 8% and 35% concentration, and promptly washing out the solution.

35. A process which comprises introducing a mixture of paper pulp and a solution of mercerizing activity between and adjacent the center of a pair of relatively rotating discs closely spaced and arranged to squeeze the fibers therebetween without abrasive action, the fibers after being dispersed and wetted with the solution being thrown from between the discs and immediately thereafter treated to reduce the activity of the solution in contact with the fibers below mercerizing activity.

In testimony whereof we affix our signatures.

FRANK H. McCORMICK.
GEORGE L. SCHWARTZ.