Feb. 24, 1953 G. A. RICHTER ET AL 2,629,295

METHOD OF MAKING PAPER

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FIG. 1.

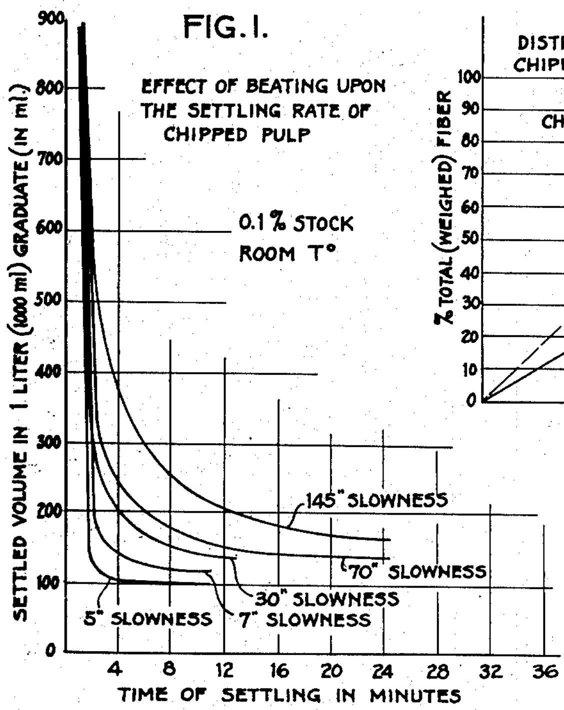


FIG. 3.

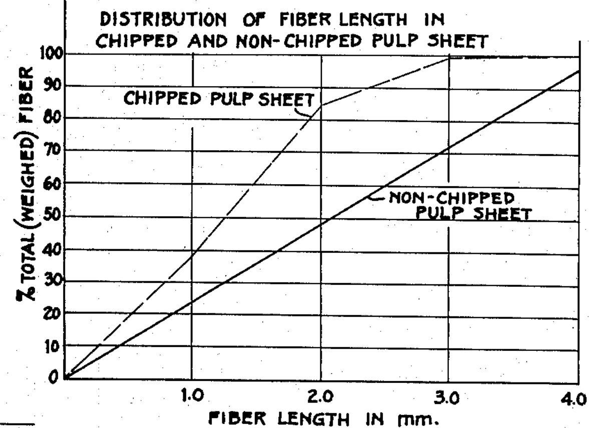


FIG. 2.

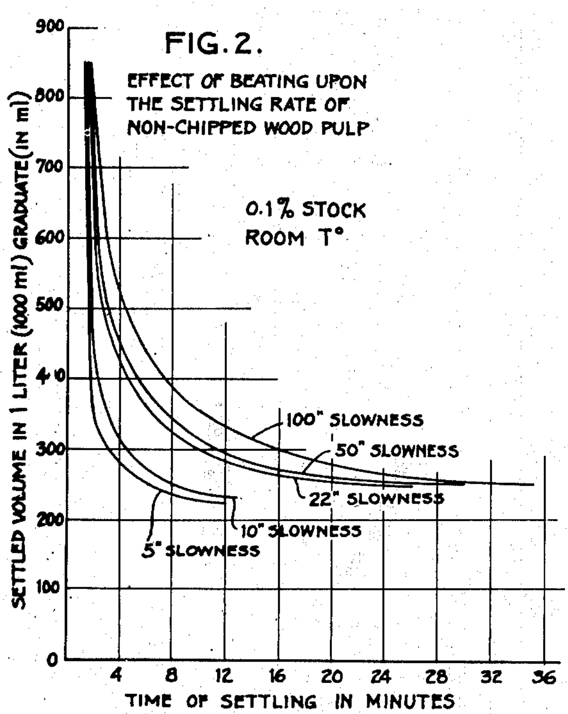
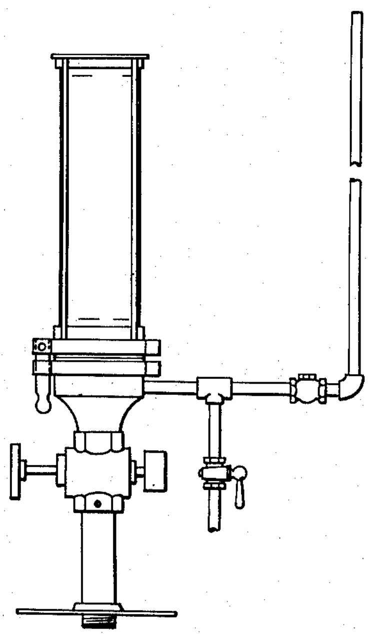


FIG. 4.



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METHOD OF MAKING PAPER

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This invention relates to a process of making paper from wood pulp in which wood pulp sheet is cut while in dry form into chips having a width of $\frac{1}{8}$–$\frac{1}{64}$ inch, which chips are dispersed in water and paper is formed from that dispersion.

Ordinarily in the making of paper, cellulose fibers are shortened by a wet operation such as by treatment in the beater or Hollander, the Jordan, or both. Shortening of fiber under these conditions is unavoidably accompanied by a partial hydration or gelling of the fiber. Hydration of the fiber gives effects which are frequently undesirable, particularly in most types of photographic paper, and therefore in prior practice in making those types of paper it has been necessary to compromise between the degree of cutting and the extent of fiber hydration.

In previous cutting operations there has been no thought of displacing the wet shortening operations of paper making by this dry comminuting procedure. In many of those previous cases in which comminution has been carried out, the fiber instead of merely being shortened has also been ground or macerated so that hydration of the fiber is not avoided.

One object of our invention is to prepare paper having a low "across the machine" wet expansion, and good flatness. Another object of our invention is to prepare paper having good formation as evidenced by uniform translucency. Further objects of our invention will appear herein.

It is characteristic of many papers upon wetting, especially on one side, that curling or cockling occurs. Such paper is not entirely suitable for wet processing or for use under humid conditions, as cockle of the paper may result. Our invention is adapted to accomplish a twofold purpose of providing paper having a minimum of wet expansion and a uniform formation or translucency throughout.

Chemical wood pulp as ordinarily prepared by the pulp manufacturer is in the form of a pulp sheet or pulp board, and the fibers are mostly in the direction in which the pulp sheet or board was laid down. We have found that if this pulp sheet or pulp board such as ordinarily employed as the starting material in paper-making processes, particularly that made up of the fibers of coniferous woods characterized by being longer than the fiber of non-coniferous wood, is cut across the machine direction of the pulp, or in other words at right angles to a large proportion of the fibers in the sheet, so that substantially all of the fibers are reduced to a length

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of not more than 3 mm. or $\frac{1}{8}$ of an inch a paper may be prepared therefrom having highly desirable properties. This is accomplished by first chipping the pulp sheet into chips whose width is $\frac{1}{8}$–$\frac{1}{64}$ of an inch, cutting across the machine direction of the pulp. This cut or chipped pulp is disintegrated and suspended in water so as to form a dispersion having a slowness of 5–100 seconds and a settling character such that a 0.1 per cent furnish settles to a volume of pulp less than 20 per cent of the original volume after 10 minutes of settling, and paper is made from that suspension. The paper obtained exhibits low "across the machine" wet expansion, good flatness, good resistance to curling; and good formation.

After the pulp has been chipped, it must be dispersed to form an aqueous mass suitable for forming paper. This may be done in the beater in which the bed-plate is adjusted to produce the desired slowness within the previously mentioned range. If the dry cutting or chipping results in sufficient shortened fibers to assure the slowness desired, no beating is necessary and, in fact, the cut fibers may be dispersed without the use of either a beater or a Jordan, such as in a propeller-type mixer. If, however, more fiber shortening is desirable, this may be accomplished by processing through a Jordan or in a beater with a minimum of hydration so that the resulting pulp slowness is between 5 and 100 seconds, such as 18, 40, 55, 65 or 83 seconds.

In the accompanying drawing, Figures 1 and 2 are graphs comparing the characteristics of beaten pulp prepared in accordance with our invention with beaten pulp which had not been previously chipped. These graphs illustrate the height of the settled pulp after settling as occurred with pulps of various slownesses, Figure 1 being directed to pulp which had been chipped as described herein and then beaten, and Figure 2 being directed to the settling heights of pulp suspensions in which pulp sheet has been beaten up without first chipping. It will be seen from these curves that the settled height and settled volume of a chipped pulp having a slowness of less than 100 seconds is less than 20 per cent of the original height or volume of the pulp dispersion after 10 minutes of settling. The pulp dispersion employed was one liter in volume and of 0.1 per cent concentration and after settling for 10 minutes, the dispersion of the pulp having "slowness of less than 100 seconds," is less than 20 per cent of the original volume of one liter.

Figure 3 is a graph in which the fiber length of the suspended fibers in millimeters is plotted against the percentage of the total weighed fiber present. It may be seen from this graph that the fibers of the non-chipped pulp sheet are fairly evenly distributed in accordance with fiber length. The fibers having a length of 0 to 1 mm., for instance, constitute one-fourth of the total mass and so on with each of the other divisions. With the chipped pulp sheet on the other hand, it may be seen that practically all of the fibers present are less than 3 mms. and the greater part of the fibers therein are less than 2 mms. in length. The proportions of the fibers of various lengths are determined by dispersing the pulp in water and determining the number of fibers of the various lengths by microscopic examination. In this way the number of fibers within the given lengths may be easily determined.

Figure 4 is a drawing of a Williams freeness tester which is employed for determining the slowness of the pulp dispersions. This tester consists of a graduated cylinder graduated from 0 to 1,000 mls. so arranged that the liquid therefrom must pass through a screen in running a freeness test. The screen employed is one having a 78 x 56 wire mesh screen. The tester is also fitted with a ball check to avoid formation of vacuum below the screen so that uniform draining of water from a pulp dispersion being tested is accomplished. The use of the Williams freeness tester in determining the slowness of a pulp dispersion will be described hereinafter.

Paper obtained in accordance with our invention with a stearic acid sizing has an "across the machine" wet expansion of less than 1.8 per cent, this value usually being within the range of 1.3 to 1.5 per cent. With paper prepared from a pulp which has been shortened only in the wet state by beating or jordanning, this paper with a stearic acid sizing has an "across the machine" wet expansion on the order of 3 per cent or possibly more. The papers referred to are those beater sized with sodium stearate and alum in accordance with the disclosure of U. S. Patent No. 1,840,399 of Gerould T. Lane. Instead of stearic acid, the paper may be sized with rosin and a similar difference in wet expansion will be observed between a paper prepared in accordance with our invention and one prepared in the customary manner.

It is possible to very considerably reduce the wet expansion of a paper prepared from a pulp which has been shortened by beating, by beater sizing the pulp with a melamine-monoureid-formaldehyde sizing or with a melamine-formaldehyde sizing as taught in U. S. Patent No. 2,548,513 of Boughton. In fact, paper may be prepared by this means having a wet expansion less than paper prepared in accordance with our invention which has not been sized with a wet-resistance-imparting sizing material. However, paper prepared in accordance with our invention which paper has embodied therein a wet-resistance-imparting sizing material such as wet-strength-imparting melamine-formaldehyde resin or melamine-monoureid-formaldehyde resin exhibits a substantial decrease in "across the machine" wet expansion over that of paper, similarly sized, prepared from a pulp which has been shortened by beating.

The term "paper of good formation" as used herein refers to paper in which the pulp fibers are uniformly distributed through the paper sheets so that a fairly uniform pattern, when viewed by transmitted light, is obtained and the cloudy, mottled effect often seen in papers is avoided. Paper of good formation (that is having a uniform translucency) is especially useful for photographic purposes.

One of the advantages of our invention is that the speeds of paper machines may be increased thereby. This is accomplished in that to obtain comparable papers, pulps of less slowness may be used in accordance with our invention giving paper of the same formation as is obtained with pulps of higher slowness where chipping has not been employed. For instance, a pulp of cellulose fiber shortened by wet preparation of 25 seconds' slowness will be equalled or exceeded in paper formation by a pulp of 19 seconds' slowness prepared in accordance with our invention. It is preferred that the pulp in accordance with our invention when ready for the machine have a slowness of 5–40 seconds, for example 8, 14, 26, 31 or 37 seconds.

The forming of the pulp-board chips which are employed for making paper, in accordance with our invention, may be accomplished by cutting the pulp board with blades or chipping knives, such as affixed to a rotating member which is rotating at highest speed. It is desirable to have a bed plate or other metal surface to facilitate the chipping operation, it being preferable that the chipping knives be so adjusted that there is a small clearance between the knives and the bed plate. The speed of feeding the pulp board to the rotating cutting member should be adjusted depending upon the speed at which the rotating cutting member is operated so as to give chips having a width of not more than $\frac{1}{16}$ of an inch, preferably approximately $\frac{1}{32}$ of an inch. For convenience the chips may be of a length of ¼–6 inches. The pulp board or pulp sheet from which the chips will be formed may be any suitable commercial form of felted pulp. The sheet may be of any width capable of operation in the machine. The thickness of pulp board which is moved toward the cutting blades should be such that these blades will quickly and satisfactorily cut and/or tear the widths therefrom. Ordinarily, it is desirable that this thickness of the pulp board be from about $\frac{3}{16}$ to about ¼ of an inch. If the pulp sheet which is used is about $\frac{1}{16}$ of an inch in thickness, obviously it will be desirable to run only three or four thicknesses of the particular pulp sheet through at the same time. Due to the stiffness of the pulp sheet or pulp board which is cut while in dry condition, there is no need for any contacting of the cutting knives with the bed plate; and for many purposes it is desirable that such contacting be avoided to avoid any contamination by metallic particles.

The pulp sheet employed may be any pulp having good paper-making qualities. For example, the pulp employed may be a sulfite pulp, a kraft pulp, or a high alpha type. The pulp sheet employed for the starting material for our invention is of fibers extracted from coniferous wood, such as spruce, hemlock, fir or pine by a cooking or digesting operation.

In the carrying out of the cutting operation, it is the best practice to operate so that a good, clean cut is obtained. In the case of some pulps additional drying of the pulp sheet is desirable to get a clean cut. For instance, it is often desirable with some types of pulp to dry it down to a moisture content of less than 1% for the best cutting. In the case of other types of pulp, clean cutting may be obtained without additional drying. As a general rule, kraft pulp is cut better when bone dry while sulfite pulp need not be dried beyond air-dry condition, for instance 5–10 per cent moisture content, for good cutting to occur.

We use sheet pulp in which the fibers primarily run lengthwise of the sheet so that the fibers are shortened. Pulp sheets as ordinarily prepared on the cylinder machine are laid down with the fibers lengthwise and these sheets are eminently suited for use in our process.

After the dry cutting of the pulp sheet has occurred, the material being obtained in the form of chips, this material is then disintegrated in water to form an aqueous suspension of the pulp. This dispersion of the pulp in water may be accomplished by various means, such as, for instance, by passing the water containing the pulp chips through a pump of a type which will disintegrate the chips, in a beater and/or Jordan providing the pulp obtained has a slowness of 5–100 seconds, preferably 5–40 seconds, and a settling rate as previously specified.

In the preparation of paper in accordance with our invention a sizing material is preferably incorporated into the pulp dispersion at some time prior to the time when it is formed out onto the wire of the paper machine. For instance, the sizing material may be rosin, hydrogenated rosin or stearic acid, which has been saponified such as with caustic soda. Ordinarily, the size may be prepared from the rosin, hydrogenated rosin or stearic acid by treating with ⅓ of its weight of caustic soda. The soap thus formed can be used in any dilution desired.

When the sizing is added to the pulp dispersion there is also added thereto some acidic material, the most commonly used material for this purpose being alum. Other acidic materials rather than alum may be used such as aluminum chloride, sulfuric acid, hydrochloric acid or the like.

In addition to or instead of the above-mentioned sizing materials the paper may be sized by incorporating in the pulp dispersion a melamine-formaldehyde resin or a melamine-monoureid-formaldehyde resin. For instance, a hydrochloric acid solution of a melamine-formaldehyde resin having wet strength-imparting properties as described in U. S. Patent No. 2,345,543 may be added to the pulp dispersion and thoroughly mixed therein prior to forming paper therefrom on the wire of the paper machine. If desired, a melamine-monoureid-formaldehyde resin having wet-strength-imparting properties may be added to the pulp dispersion prior to paper formation.

After the pulp has been throughly disintegrated so as to form an aqueous suspension and a sizing material has been incorporated therein, it is formed onto the wire of the paper machine so as to form a hard, dense paper comparable in character as to denseness, or even more dense, than paper ordinarily prepared. The paper which is prepared in accordance with our invention should preferably have a weight of 3 to 6 pounds per 1,000 square feet per $\frac{1}{1000}$ of an inch thickness. Due to the fact that the pulp in accordance with our invention occupies less volume upon settling than wet-shortened pulp fibers, the obtaining of a dense sheet is not difficult. Paper in accordance with our invention is especially suitable for use as the base of photographic paper.

As the coating compositions in making photographic papers are aqueous, paper which is not affected by wetting and then drying is of value for this purpose.

Photographic papers in use are subjected to aqueous baths such as in developing, printing and washing. The property of low wet expansion which characterizes our paper is especially useful in photographic paper in which flatness and the absence of cockling and curling is desirable.

The following examples illustrate our invention:

Example 1

A pulp sheet of high alpha sulfite pulp obtained from Western Hemlock wood by digesting and refining, is chipped by running a roll of the pulp sheet longitudinally through a chipping mechanism which sharply shears strips of approximately $\frac{1}{25}$ inch width and ¾ inch long therefrom, thus cutting across the fibers of the sheet. Six hundred pounds of this chipped pulp are suspended in 1100 gallons of water in the beater, the pulp mixture being mixed with 10 pounds of sodium stearate size, 45 pounds of corn starch and 15 pounds of aluminum sulfate, the materials being added to the water in the beater in the order given. After the beating takes place for only a sufficient time to disperse the pulp in the water and to condition the pulp for forming paper, it is transferred to the paper machine, such as through a Jordan in which the knives are of sufficient sharpness and of proper design such that excessive hydration does not occur. The resultant stock having a slowness of 30 seconds is then processed over the paper machine in the conventional manner, to form a dense sheet of paper. The resulting paper shows good formation and less "across the machine" wet expansion than is exhibited by corresponding paper prepared from the pulp beaten or jordanned to shorten the fibers rather than by a dry chipping operation.

Example 2

A pulp sheet from kraft high alpha pulp obtained from Northeastern Spruce wood by digesting and refining is chipped by running a roll of the pulp sheet longitudinally through a chipping mechanism which sharply shears strips of approximately $\frac{1}{32}$ of an inch width and ¾ inch length therefrom, thus cutting across the fibers of the sheet. Six hundred pounds of this chipped pulp are suspended in 1100 gallons of water in the beater avoiding abnormal hydration. The pulp suspension is mixed with the following materials added in the order given:

5.6 pounds of sodium aluminate
9.6 pounds of sodium stearate
3.0 pounds of gelatin
45.0 pounds of cooked corn starch
4.5 gallons of hydrochloric acid (24%)

The beating is only continued for a sufficient time to assure good dispersion of the pulp and to condition the pulp for the formation of paper. A hydrochloric acid solution of an acid-aged melamine-formaldehyde resin as described in Wohnsiedler Patent No. 2,345,543 is then added to the stock in an amount that the resin constitutes ½ per cent of the weight of the bone dry pulp.

The resultant stock having a slowness within the range of 25 seconds is then processed over a paper machine in the conventional manner. A paper of good formation is obtained having "across the machine" wet expansion superior to that of papers of like kind in which beating is depended upon rather than dry chipping for shortening of the fibers of the pulp.

Example 3

A pulp sheet of high alpha sulfite pulp is chipped as described in the preceding examples to produce chips of 1/32 of an inch width and 3/4 inch length and is then suspended in water by means of a beater as previously described. There is added to the pulp dispersion approximately 1% of hydrogenated rosin size and approximately 1% of aluminum sulfate, the percentages being based upon the bone dry weight of the pulp. The hydrogenated rosin size is prepared by saponifying hydrogenated rosin with approximately 1/3 its weight of caustic soda. After the pulp has been thoroughly dispersed in the water forming a dispersion of 20 seconds and is in condition for forming paper, it is passed to the paper machine preferably through a Jordan in which the knives have been so adjusted that little or no hydration occurs therein. The resultant stock is then processed over the paper machine in the conventional manner.

Example 4

Paper is prepared in the same manner as in Example 2 except that a melamine-monoureid-formaldehyde resin in solution in hydrochloric acid is added to the stock in an amount 1/2% of the weight of the bone dry pulp instead of the melamine-formaldehyde resin employed therein. The paper sheet is made on the machine in the customary manner.

Example 5

Paper is prepared in accordance with the preceding example. It is then dried and a silver halide-gelatin photosensitive emulsion is applied to one surface of the paper, preferably after first coating the paper with a baryta layer consisting of gelatin and barium sulfate as is known in the art. The photosensitive emulsion is applied under darkened conditions to prevent fogging of the emulsion. A photographic paper is obtained which is less affected by processing solutions and washing than a corresponding paper prepared in the manner of the prior art.

The wet expansion of paper and its shrinkage upon drying is determined by a well known instrument known as a "Schopper Apparatus for Testing the Expansion of Paper under the Influence of Moisture" manufactured by Louis Schopper of Leipzig, in which changes in dimensions of the paper cause a pointer to move over a graduated scale. Further description of the apparatus for measuring the expansion and contraction of paper is given in an article by Griffin in the Paper Trade Journal of August 4, 1927.

The slowness of paper pulp as used herein is determined with a Williams freeness tester as illustrated in Figure 4 of the drawing in accordance with the following procedure: One pound of bone-dry pulp is suspended in 20 pounds of water at 70° F. in some device in which the fiber is brought into suspension without any hydration occurring. A Valley beater may be employed for this purpose without using any weight on the bed-plate lever. Where the pulp is already in suspension it may be diluted to correspond to a suspension of 1 pound of fiber in 20 liters of water and used directly in the following test: A 44–45 cc. sample of slush as prepared above, is diluted to 1000 cc. with water, all at a temperature of 22.5° C. (70° F.) Water having the same temperature is added to the Williams freeness tester up to the 0 cc. mark and then the 1000 cc. mass of water and fiber is poured into the graduated cylinder of the tester. As soon as the air bubbles (formed by the pouring) have risen, the drainage valve is turned and the time interval for the drainage of the 1000 cc. of water from the suspended fiber is obtained. This value in seconds is the slowness of the pulp.

The settling test is carried out by taking a dispersion of pulp in water, diluting with water sufficiently to obtain a stock or furnish of 0.1 per cent concentration, placing one liter of that pulp suspension in a one-liter graduate and observing the settled volume of the pulp at selected time intervals, for instance, every two or three minutes. The selection of this time interval will depend upon the rapidity at which the curve is changing direction at any particular point. After these observations are taken, curves are then made from the data obtained therefrom as illustrated by Figures 1 and 2 of the drawing attached hereto.

It is to be understood that our invention is primarily directed to low wet expansion paper and its preparation by means of the chipped pulp described herein. A low wet expansion paper may be prepared in which chipped pulp is not 100% of the pulp used, i. e., some non-chipped pulp may be mixed therewith without impairing the low wet expansion imparting properties of the chipped pulp. Such a modification is considered as being within the scope of our invention as defined by the appended claims.

We claim:

1. A method of preparing paper which consists in transversely cutting a dry sheet of coniferous wood pulp, the fibers of which mainly run lengthwise, at intervals of approximately 1/32 of an inch, thereby forming chips of that approximate width, agitating the chips in water to disperse the pulp fibers therein and to form a suspension having a slowness of 5–100 seconds by the Williams freeness test, providing the suspension with sizing chemicals and preparing paper from the pulp suspension while maintaining the slowness within 5–100 seconds whereby paper of good flatness, low wet expansion and uniform formation is obtained.

2. A method of preparing paper which consists in transversely cutting a dry sheet of coniferous wood pulp, the fibers of which mainly run lengthwise, at intervals of approximately 1/32 of an inch, thereby forming chips of that approximate width, agitating the chips in water to disperse the pulp fibers therein and to form a suspension having a slowness of 5–100 seconds by the Williams freeness test, providing the suspension with malamine-formaldehyde resin paper size and preparing paper from the pulp suspension while maintaining the slowness within 5–100 seconds whereby paper of good flatness, low wet expansion and uniform formation is obtained.

3. A method of preparing paper which consists in transversely cutting a dry sheet of coniferous wood pulp, the fibers of which mainly run lengthwise, at intervals of approximately 1/32 of an inch, thereby forming chips of that approximate width, agitating the chips in water to disperse the pulp fibers therein and to form a suspension having a slowness of 5–100 seconds by the Williams freeness test, providing the suspension with sodium stearate paper size and preparing paper from the pulp suspension while

maintaining the slowness within 5–100 seconds whereby paper of good flatness, low wet expansion and uniform formation is obtained.

4. A method of preparing paper which consists in transversely cutting a dry sheet of coniferous wood pulp, the fibers of which mainly run lengthwise, at intervals of approximately $\frac{1}{32}$ of an inch, thereby forming chips of that approximate width, agitaing the chips in water to disperse the pulp fibers therein and to form a suspension having a slowness of 5–100 seconds by the Williams freeness test, providing the suspension with hydrogenated rosin paper size and preparing paper from the pulp suspension while maintaining the slowness within 5–100 seconds whereby paper of good flatness, low wet expansion and uniform formation is obtained.

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