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TREATMENT OF CELLULOSIC PULPS

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The present invention relates primarily to a fiber board product of a greater density and strength than has hitherto been possible. The invention, moreover, also includes certain process steps that involve the control of the relative freeness of suspensions or furnishes of partially hydrated cellulosic fibers, particularly reduction of the slowness of hydrated pulps without impairing the advantages resulting from the hydration of the fibers. It also involves process features involving the precipitation of negatively charged colloidal particles upon similarly charged suspended cellulosic fibers.

All of these features are so interrelated as to constitute one comprehensive inventive concept, leading to the production of fiber boards, of the conventional insulating board type, but endowed with improved strength, stiffness, density, and with other valuable properties.

In the manufacture of fiber insulation boards, the fibrous pulp which is made of wood, bagasse, straw or other similar materials, is suspended in water, usually 99% water or more. The distended fibers are thereafter deposited on a wire screen either by gravity or by suction, by allowing the water to be drawn away through the deposited fibrous mass, thus resulting in a wet mat or felted mass of fibers. The wet felted mass is then pressed by rollers or the like, usually between wool felts or wire screens, to eliminate all the water possible and to produce a compacted felted board usually about ½" thick or more, which when dried forms the insulation or building board commonly used in building construction as a sheathing, interior wall, or ceiling covering.

In this type of manufacturing process it will be evident that the drainage rate of the fibrous pulp is most important, as otherwise only a very thin paper-like sheet can be formed.

The strength of the resulting board depends largely on the preparation of the pulp. If the fibers are well beaten they hold more water and become "hydrated," as it is known in the art. Hydrated pulp, however, becomes very slow draining, so that it is formed in thick sheets with difficulty, cannot be pressed to any degree without deforming or "crushing" the board, and is difficult to dry. The dried product, however, is stronger, harder and more lumber-like. Hydrating pulp to any degree, besides causing difficult processing, adds materially to the expense of preparing the product.

Normal fiber board pulp, made in accordance with the usual methods, can only be slightly hydrated if an economical process is to be maintained.

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Due to the springy nature of the fiber when only slightly hydrated, it cannot be pressed to higher densities than about 17 to 19 pounds per cubic foot, and therefore the product thus produced is limited to relatively soft insulation board products.

Accordingly, it is one of the objects of the present invention to overcome the slowness of a hydrated pulp, so that denser and stronger products may be made, using the standard type of equipment now used in the paper and pulp industry.

A further object is to produce a fiber board containing therein distributed lime or other alkaline earth hydroxides.

A further object is to provide a process for making such a board.

A further object is to provide a process by means of which rubber or resin particles may be incorporated with a paper furnish, and deposited upon the fibers thereof.

Other objects will appear from the further and more detailed description which follows.

I have discovered that if an alkaline earth hydroxide, such as lime hydrate (high calcium) or quicklime (since it slakes to lime hydrate), or the hydroxide of barium or strontium, is added to a cellulosic fiber suspension the drainage rate or "freeness" of the suspension is increased, making it possible to use a more hydrated pulp without having trouble with formation of the paper sheet or board mat on the forming machine; the water will drain away more easily on the screens or felts and the fibers will compact to a thinner sheet under the same pressing conditions. In the case of pulp mats, these will shrink more in the drying operation and the resultant dry mat will have a higher density and greater strength than an untreated mat subjected to the same operating conditions.

Although the scope of the invention is intended to include all amounts of alkaline earth hydroxides, which when added to a suspension of cellulosic fibers in any way will give any or all the above results to any degree, the preferred method is to predisperse superfine high calcium quicklime in water, and allow it to hydrate. When, for example, making pulp boards such a dispersion is added to the hot (110°–120° F.) fiber suspension or furnish at such a rate that the amount of undissolved lime hydrate in the furnish will be at least about 1% of the dry pulp, and at such a point in the process that the mixing be-

fore the mat is formed will be sufficient to give good dispersion of the alkaline earth hydrate. Hot slurry is preferred, in case of calcium hydroxide, because this hydrate is less soluble in hot water, which hence means less loss. In general about 10% of the alkaline earth hydroxide will be the maximum that needs to be used; although lower percentages will still produce decided increases in freeness. The addition of the alkaline earth hydrate has been found to increase the tendency of the furnish to develop foam to a varying degree, depending on the amount of rosin or rosin acids present; with pine pulp showing a greater tendency to foam than others. To reduce this tendency, an anti-foaming agent may be added with the alkaline earth hydrate and I have discovered that an agent must be used which does not react with the alkaline earth hydrate in any way, as this would have an adverse effect upon its action. Since alkaline earth hydrates are cationic in nature, it is preferred that a cationic type of anti-foaming agent be used, as these are less likely to be reactive toward the lime hydrate. Hydrochloric, acetic or other acid salts of long chain amines have been found satisfactory.

This type of material, being cationic in nature, will act as an anti-foaming agent against any foam caused by an anionic material. It is also a wetting agent, and would tend to destroy the sizing of the mat except that in the drying of the wet mat the acid component of the amine salt is driven off, leaving the amine, which is water-resistant. The acetic acid salt of such an amine breaks down more easily under heat and therefore is preferred over the others.

Rosin size is not well adapted to this process as a material to give the board water resistance. I prefer to use, instead, a cook starch-wax type of size having the wax emulsified in the cooked starch.

A size, made up as follows, may be used:

	Parts
Pearl starch	100
Water	2000
Cooked to 195° F. and	
Paraffin wax, emulsified therein	200

With this sizing material is it possible to obtain satisfactory water resistance, comparable with regular insulation board even in the alkaline water caused by the addition of the lime hydrate. This in itself is beneficial, since the board will not deteriorate as fast on aging as it would if it were acid.

The action of the lime or other alkaline earth hydrates is not fully understood, but it is believed that their action is similar to that which is obtained when a positively charged colloid is added to a colloid of opposite charge, namely a phenomenon of co-precipitation. The more hydrated the original pulp, the closer it approaches the properties of a negatively charged colloid and the more attraction it has for water, which causes the slower drainage rate or lower "freeness." When the positively charged alkaline earth hydrate is added there occurs a co-precipitation action between it and the pulp. Since the water-attractive charges of the pulp are neutralized by the hydrate, the "freeness" or drainage rate increases. It has been found that an excess of hydrate may couse a decrease in freeness due to the "filter plugging" action of the felted fibers. In the formed or felted mass the attraction between the fiber and water of the hydrated pulp has been changed to an attraction between the pulp and alkaline earth hydrate. This mutual attraction of the co-precipitated mass of fibers and hydrate reduces the tendency of the fibers to spring back after being pressed to a certain thickness. In the drying operation the same attraction pulls the mass closer together, producing a higher density in the ultimate product. The increased strength of the dried product is also due to this fiber-to-hydrate-to-fiber bond or attraction.

The process of the present invention may be applied to the usual method of making pulp insulation board, using as an added ingredient the alkaline earth hydroxide such as barium hydroxide, strontium hydroxide, and more particularly, calcium hydroxide. Because of its cheapness, the latter is preferred; and the further description is devoted to a process in which lime forms the source of the hydroxide. This may be either in the form of quick-lime (CaO), which is slaked in the course of the process, or in the form of so-called "hydrated lime" which is obtainable in dry form as an almost impalpable powder. A high calcium lime is preferred. When using other alkaline earth hydroxides, equivalent quantities should be used to produce the same degree of alkalinity. These quantities can be readily calculated from the atomic weights of barium and strontium as compared with that of calcium.

The process may be carried out as follows:

A furnish containing 1500 parts by weight (dry basis) of ground wood fibers, such, for example as cottonwood or willow, at a concentration of 1% (i. e. suspended in 150,000 parts of water) is worked, as in a beater or similar instrumentality, partially to hydrate the fibers. The furnish, preferably warmed to a temperature of about 110° F., is then given an addition of about 360 parts of high calcium lime hydrate ($Ca(OH)_2$), which latter is preferably first mixed with sufficient water to provide a cream-like slurry. About 1,000 parts of water will suffice. The addition of the hydrated lime will increase the apparent "freeness" of the furnish by an amount proportioned to the degree of hydration of the pulp. Either before or after the addition of the lime, a suitable amount of the above-mentioned starch-wax emulsion may be mixed with the furnish to yield about a 2% to 3% paraffin wax content in the finished board. The furnish is then fed to the headbox of a suitable forming machine and formed into a wet mat, from which the water is expressed by means of rolls, which act upon the mat through the intervention of a suitable traveling screen and wool felts. A regular Oliver board former forms an excellent apparatus in which to carry out the process; but it is not limited to the use of this particular machine, as Fourdrinier machines and other well known board formers may be used. As all these are so well known in this art, a detailed description thereof would serve no useful purpose.

After the free water has been expressed from the wet mats, they are cut into large sheets by the usual transversely traveling cutters, and elevated by the usual tipples into a multi-deck drier, such as a Coe drier, or the like, and dried to about 5% moisture content. They may then be cut into any desired dimensions.

The results obtained by the addition of the lime to the furnish are drastic and far reaching. Thus the freeness is increased, the board is thinner, much denser and has a greater strength. These

results will vary depending on the amount of lime hydrate added; the method of addition; the temperature of the water slurry; the degree of dispersion of the hydrate particles and the amount of hydration of the pulp.

If the pulp were more hydrated, then the untreated freeness would be lower, but the treated freeness would be approximately the same, thus indicating more strongly the "freeing" action of the lime hydrate.

I have found that slaked quicklime or lime hydrate may be used with any suspension, emulsion or colloidal solution of a material, which has a negative particle charge, to flocculate the material and cause it to be retained by the pulp. In this way the strength and other properties of a lime treated mat may be altered in many directions, i. e., higher water resistance, more tough, harder, or more elastic, depending on the type of material added with the lime hydrate.

The mechanism of the above process is similar to the action of lime on pulp. In this case the negative charge of the emulsified particle and the pulp is neutralized by the lime so that they no longer repel each other, but rather are joined or attached by the lime hydrate.

In this way a negatively charged rubber emulsion may be used with lime in the pulp treatment to increase the elasticity or toughness of the mat. Various negatively charged resin colloids may be used to increase hardness and strength, and so on; as long as the material particle has a negative charge, the lime hydrate will neutralize the charge and cause the retention of the particle on the pulp. If wood pulp is hydrated into a state approaching a colloidal solution, and is then used with lime in the treatment of pulp, the same action occurs and the strength of the resultant mat is increased considerably. This makes it possible to refine the base pulp a relatively small amount, add approximately 4% of colloidalized pulp with lime, and obtain a mat considerably stronger. The lime hydrate also resists the tendency of the colloidal pulp to reduce the pulp freeness.

In the actual use of this lime hydrate treatment in the wet forming process, any type of cellulose fiber may be used, but the degree of action of the lime depends on the point to which the fiber has been hydrated. If there is no hydration, the action as described will be negligible. For purposes of definition in connection with the disclosure of the present invention, the term "hydration" means not plain refining or reduction to individual fibers, but further beating or mechanically working to a state in which the hydrogen bonds of the cellulose or lignocellulose micelles of the fibers are broken, giving unsatisfied charges or valences which have an attraction for water and give the pulp a gelatinized appearance in varying degree, depending on the amount of hydration.

In the use of the lime hydrate treatment in the wet-forming process, the preferred method is to predisperse 1 part of high calcium quicklime in 3 parts of cold water and allow it to hydrate, after which it is diluted with four additional parts of water. This dispersion may then be added to the pulp slurry at any point from the thickeners to the forming machine. Addition of the lime at the headbox is preferred.

Any sizing material, for example, the starch-wax size described previously, may be added with the lime hydrate or separately at any other point, from the thickeners to the forming machine. Good mixing of the size and hydrate with the pulp should be insured before the mat is formed.

It is preferred to add the lime hydrate slurry to the pulp at such a rate that the undissolved portion of hydrate retained by the fibers will be between 5% and 10% of the dry mat weight.

If it is desirable that a foam depressant be added as previously mentioned, it may be added with the size, with the lime hydrate, or at any other suitable or convenient point. The amount required is a few tenths of 1 per cent.

In the formation of the mat on the forming machine cylinder or wire, the lime hydrate treatment gives the pulp the property of losing its water more easily and compacting to a thinner mat having a lower water content. In the wet pressing operation it is also possible to compact the pulp to a much higher density and a lower water content than if it had not received the hydrate treatment. In the drying operation the mat shrinks to a greater degree and the dried mat, containing approximately 6% of lime, will have a density of from 25 to 28 pounds per cubic foot as compared with a density of 16 to 17 pounds per cubic foot for a mat made of the same pulp but without the lime hydrate treatment, but otherwise under the same forming conditions.

The following table gives a comparison of the test results on boards made on commercial equipment. Both mats were formed and made under identical conditions except that the pulp for No. 1 was treated with the lime hydrate slurry, while No. 2 had no lime added. Transverse strengths were found using a 12" span.

Table I

Percent CaO	Thickness	Density	Transverse Strength	Modulus Rupture	Tensile Strength	Hardness Kg/.01" Depression
	Inches	*Pounds/ft.*^{3}	*Pounds*	*Pounds*	*P. s. i.*	
No. 1, 5.54	.473	25.4	33.5	900	544	30
No. 2, 0	.521	16.7	12.8	283	165	11

If the pulp used to form these mats had been more "hydrated" the results would have shown higher values.

Further to illustrate the advantages obtained by the practice of the present invention, boards were made up both with and without the use of the lime, and the following results obtained:

	Without Lime	With Lime
Freeness increase, cc.	none	20
Drying shrinkage, percent	12.3	20.2
Thickness when dry, inches	0.467	0.367
Density, dry, lbs./cu. ft.	17.8	27.7
Modulus of Rupture, lbs.	339	998
Transverse strength, lbs.	24.6	44.8

It will be noticed that the modulus of rupture increased by about 300 per cent. This is also reflected in the large increase in transverse strength and the far greater density. Boards made by this process can therefore be used under conditions where ordinary insulation boards would prove inadequate.

To illustrate the effect of lime in showing a larger increase in freeness when the pulp is "hydrated" to a greater degree and to show the similar effect of the other alkaline earth hydroxides, boards were made from pulp having a Schopper freeness, at 1% consistency, of 600 cc. which showed it to be relatively "unhydrated" and also from the same type of groundwood pulp which had been further beater "hydrated" to a freeness of 350 cc. The two types of pulp were used with and without the lime treatment. Equivalent quantities of barium hydroxide and strontium hydroxide were used with the hydrated type of pulp. The following results were obtained:

Table II

Type of Mats	Original Freeness	Freeness Increase	Dry Density	Modulus of Rupture
	Cc.	*Cc.*	*Pounds/ft.*^{3}	*Pounds*
Unbeaten Pulp	600	None	16.6	282
Unbeaten Pulp +$Ca(OH)_2$	600	65	22.3	635
Beaten Pulp	350	None	19.2	615
Beaten Pulp +$Ca(OH)_2$	350	175	26.2	1,205
Beaten Pulp +$Ba(OH)_2$	350	175	25.3	820
Beaten Pulp +$Sr(OH)_2$	350	175	22.5	840

It will be noticed that the lime caused a freeness increase of only 65 cc. on the unbeaten pulp as against 175 cc. with the beaten pulp. Both barium and strontium hydroxide gave the same freeness increase as lime on the beaten or hydrated pulp. All three alkaline earth hydroxides show an increase in modulus of rupture and density with the lime giving the highest values.

In order to show the suitability of a lignin-free or pure cellulosic type of pulp in this process and also to emphasize the need for hydration as described above, a series of boards were made of bleached sulfite pulp. In the following data, by swollen sulfite is meant bleached sulfite pulp which had been treated for 24 hours in a 15% solution of NaOH so that the fibers became swollen with absorbed water, thus giving chemically hydrated pulp without actual fiber rupture. This pulp was then washed free of caustic before using.

The same technique was employed as when making boards from a lignocellulosic pulp. The results are shown on the subjoined Table III.

Table III

Type of Mat	Original Freeness	Freeness Increase	Dry Density	Modulus of Rupture
	Cc.	*Cc.*	*Pounds/ft.*^{3}	*Pounds*
1. Sulfite Fiber	720	----------	26.3	279
2. Sulfite Fiber+$Ca(OH)_2$	720	none	27.4	270
3. Sulfite Fiber Beaten From 720 cc. Freeness To 200 cc. Freeness+ $Ca(OH)_2$	200	200	43.7	3,315
4. Sulfite Fiber Beaten as above Without $Ca(OH)_2$	200	----------	41.9	2,425
5. Swollen Sulfite+$Ca(OH)_2$	720	----------	13.5	----------
6. Swollen Sulfite Fiber Beaten from Freeness of 720 cc. to 450 cc. Freeness+$Ca(OH)_2$	450	100	38.0	2,060

It will be noticed that the lime hydrate had no effect on the plain unbeaten sulfite fiber, giving no increase in strength or freeness, but in the case of #3 when the fiber was beaten, thus rupturing the fiber cell, to a Schopper freeness at 1% consistency of 200 cc., the lime hydrate increased the freeness by 200 cc. and gave a modulus of rupture of 3,315 pounds. In the case of #5 the lime hydrate had no effect on the fiber which had been hydrated chemically without fiber rupture, but in the case of #6 when the same type fiber was beaten to a freeness of 450 cc., the lime hydrate addition increased the freeness to 550 cc. and resulted in a board having a modulus of rupture of 2,060 pounds.

It can be seen from these results that a lignin-free or pure cellulosic pulp may be used in this process if it is hydrated as described previously by actually rupturing the fiber cell.

The examples given show the unusual results obtained with fiber boards used for structural purposes. Obviously the same type results may be expected in paper or other fiber products produced by wet-felting fibers, pressing and drying in a manner similar in principle to the methods disclosed. Certain classes of paper products, such as "glassine" and fiber sheet cartons, where high strength coupled with dense structure and stiffness is important, can be vastly improved and made more economically using the methods as specified in this disclosure.

While the process has been described in connection with wet ground wood pulp made from cottonwood or willow, it is also applicable to pulps of other origins, which are hydrated to any degree as described previously, such as pine, poplar and other woods, which may have been pulped by boiling with water either at atmospheric or superatmospheric pressure, and with or without the aid of pulping chemicals. It is also applicable to the type of pulps obtainable by the so-called "explosion" process in which wood chips are heated in high pressure steam and then suddenly released into atmospheric pressure surroundings.

From the above disclosure it will also be apparent that the use of the alkaline earth hydroxides enables a determination of the felting qualities and true hydration of any cellulosic pulp, irrespective as to its intended utilization. The "freeness" or "slowness" of a given pulp is determined by a number of factors, only one of these being an indication of true hydration. Thus if a furnish contains numerous fine particles which tend to close up the openings in a Schopper-Riegler Freeness-tester, there will be given a false indication of the actual hydration, and the stock will be adjudged slower than it really is. Such fine fibers, while slowing up the stock, do not contribute materially to increased strength and density of the product made therefrom. On the

other hand, if the slowness (i. e. lack of freeness) of the stock is due to real mechanical hydration, i. e. fibrillation of the cellulosic fibers which causes them to adsorb and hold water, then this will be a fairly accurate indication of the expected strength of the product to be made whether it be paper, pulp, or fiber board.

By following the teachings of the above disclosure one can readily determine the actual hydration of the pulp. Thus it has been found that by adding an alkaline earth hydroxide, such as calcium hydroxide (lime hydrate) to a hydrated pulp, the measured freeness of the pulp will be increased by an amount that is proportional to the degree of true hydration.

By measuring the freeness of a pulp by the hereinabove described method, a result is obtained which is a measure of the sum of the effect of the slowing action of the surface area of the pulp fibers and the water attraction of hydrated portion. This will give a certain freeness value. If now a sample of the same pulp is treated with calcium hydroxide (say 5% thereof on the basis of the dry pulp at 1% consistency), and then tested for freeness, it will be found that that part of the original slowness which was occasioned by the hydration is neutralized, as a result of which the apparent freeness of the pulp will increase by an amount that is proportional to the degree of the true hydration of the pulp. Hence the greater the increase in freeness caused by the presence of the alkaline earth hydroxide, the greater must have been the degree of hydration of the pulp that was tested. It follows, also, that the greater the degree of hydration, the greater will be the felting strength of the fibers, and hence they will produce a stronger and denser product, such as paper or board.

As an example of the utilization of this method of testing the hydration of a pulp one may proceed as follows:

The freeness of a certain pulp, as measured on a standard Schopper-Riegler tester was found to be 600 cc. Another sample of the same pulp, after treatment with calcium hydroxide, as hereinabove explained, then had a freeness of 665 cc., which is an increase of 65 cc. A mat made from such a pulp had a modulus of rupture of 282. The same pulp was then mechanically worked (beaten) so as to hydrate it to a freeness of only 350 cc. A sample of the thus beaten pulp was treated with calcium hydroxide and then tested for freeness, which now was found to be 525 cc., or an increase of 175 cc., as compared with an increase of only 65 cc. in the case of the unbeaten pulp. A mat made from the beaten pulp had a modulus of rupture of 615, or better than twice that of the unbeaten pulp. The degree of hydration was thus also indicated to be more than twice that of the original, unbeaten pulp.

There is thus provided a novel and very useful method for discriminating between slowness due to shortness of fiber and slowness due to real hydration; something which has not been possible by so simple means in the past.

Accordingly, and reserving to myself the reasonable adoption of equivalents and the use of the usual mechanical instrumentalities used in this art, I claim:

1. Process of making fiber board which comprises suspending ground wood fibers in water to form a fiber furnish, mechanically working said furnish to hydrate the fibers therein, adding enough lime, within the range of from about 1% to about 24% by weight of the dry fibers, to increase the freeness of the furnish by from about 65 cc. to about 175 cc. over its original freeness, forming the resulting furnish into a wet mat, compressing the latter to express free water therefrom, and drying the mat.

2. Process of making fiber board which comprises adding to a furnish containing hydrated lignocellulosic fibers a sufficient amount of an alkaline earth hydroxide, within the range of from about 1% to about 24% by weight of the dry fibers, to increase the freeness of the furnish by from about 65 cc. to about 175 cc., and forming a compressed mat therefrom, and drying the same.

3. Process of making fiber board which comprises adding to a furnish containing hydrated lignocellulosic fibers a sufficient amount of calcium hydroxide, within the range of from about 1% to about 24% by weight of the dry fibers, to increase the freeness of the furnish by from about 65 cc. to about 175 cc., and forming a compressed mat therefrom, and drying the same.

4. Process of making high density high strength fiber insulation board which comprises suspending ground wood fibers in water to form a furnish containing partially hydrated fibers, adding, within the range of from about 1% to about 24% on the weight of the dry fibers, of an alkaline earth hydroxide sufficient to increase the freeness of said furnish by about from 65 cc. to about 175 cc., forming a wet mat from said furnish, compressing the mat to express free water therefrom, and drying the resulting mat.

5. Process of making fiber insulation board which comprises grinding raw wood in the presence of water to form a furnish having a Schopper-Riegeler freeness of from about 350 cc. to about 600 cc., adding enough of an alkaline earth hydroxide thereto, within the range of from about 1% to about 24% on the dry weight of the fiber, to increase the freeness of said furnish by from about 65 cc. to about 175 cc., forming a wet mat from the furnish, expressing free water from said mat, and drying the same.

6. A fiber insulation board consisting of cellulosic fibers and from about 1% to about 24% by weight of an alkaline earth hydroxide, said board having a density of about 25 to 45 pounds per cubic foot, a modulus of rupture of about 900 to 3300, and a tensile strength of about 544 pounds per square inch.

7. A fiber insulation board consisting of lignocellulosic fiber and from about 1% to about 24% by weight of calcium hydroxide, said board having a density of about 25 to 45 pounds per cubic foot, a modulus of rupture of about 900 to 3300, and a tensile strength of about 544 pounds per square inch.

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