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PROCESS OF MAKING LIGNO-CELLULOSE PULPS FROM STRAW

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The present invention relates to a process of producing pulp from fibrous ligno-cellulose materials. The process is characterized by a mechanical defibrating treatment carried out at temperatures above 212° F. and under steam pressure, the treatment being chemically supported by acidifying the raw material previous to the mechanical defibration.

The invention has for its object to produce a method suitable for the production of inexpensive pulps, especially from straw from cereal plants, such as wheat, rye, corn, rice, etc., grasses, bagasse, flax straw, bamboo and the like.

In the manufacture of mechanical pulps from various ligno-cellulose materials a continuous method for mechanical defibration at elevated temperatures and under steam pressure has found a rather extensive application. According to this method the fibrous material is mechanically defibrated in a closed system and under steam pressure after having been heated to a temperature of above 212° F.

It has also been suggested to produce chemical and semi-chemical pulps by digesting ligno-cellulose materials with some of the usual cooking methods, e. g. the alkaline sulphate or the acid sulphite processes, and by combining this chemical treatment with mechanical defibration at elevated temperatures.

For the production of paper and board pulp from straw and similar annual plants alkaline digesting liquors, for example alkaline or lime lye are generally employed. Thus, the sulphate and soda methods employed in the production of cellulose from wood have met with extensive application for the production of higher grades of pulp from straw. The sulphite method has also been tried although to a lesser extent. Generally, however, straw pulp is produced by digesting the raw material in caustic soda-lime lye or with lime lye only, whereby a relatively coarse stock is obtained which after further refining is employed for strawboard and the corrugated sheet in corrugated boxboard.

It is likewise known to produce straw pulp by the above-mentioned mechanical high-temperature defibration without the addition of chemicals. The fibre stock obtained in this way is stiffer and coarser than the pulp obtained by the method according to this invention and cannot advantageously be refined to a paper and board pulp of better quality.

The method according to the present invention is based on the discovery that the mechanical defibration at temperatures of over 212° F., preferably 270–380° F., is highly improved and accelerated in the presence of small quantities of certain solutions which under the prevailing temperature conditions have an acid reaction. These solutions, which are referred to below as acids, may consist of solutions of mineral acids, such as sulphuric, hydrochloric or nitric acids, certain organic acids, such as acetic acid or salts, e. g. sodium bi-sulphate, sodium bi-sulphite or aluminium sulphate.

The purpose of the addition of these solutions acting as acids is not to produce a more or less extensive dissolution of the raw material, as is the case with the methods of chemical treatment customarily employed, where a dissolution of the lignin-containing constituents of the vegetable material is aimed at in order thereby to produce or at least facilitate a separation of the fibres of the material. Hitherto it has generally been assumed that in producing fibre pulp from fibrous ligno-cellulose materials such chemical agents should be employed which have a dissolving effect on the lignin constituents of the material. In the method according to the present invention chemical agents are not employed, which have this effect, but agents which rather have a tendency to act on the hemi-cellulose constituents of the material. Surprisingly enough it has shown itself that at elevated temperatures even very small quantities of the substances employed in the method according to the present invention to a very large degree facilitate the mechanical treatment of fibrous materials, especially of straw and similar, to a greater extent non-lignified vegetable materials, but also of wood from coniferous and deciduous trees. The reason for the required quantity of chemicals being comparatively slight can possibly be explained by the fact that the hydrolysing effect of the agents used in the present method is limited to the membraneous substances having a cohesive effect between and within the fibres and which only constitute a slight portion of the entire woody material.

It has shown itself that the yield of the fibrous raw material when treated with the method according to the present invention is nearly the same as when only a high temperature mechanical defibration without chemical treatment is used, since also in this case a certain diminution of weight occurs dependent upon the dissolution of water-soluble constituents. Thus, it has not been possible to establish with certainty whether the chemical treatment according to the inven-

tion causes any appreciable dissolving effect on the vegetable matter.

Therefore it may be assumed that the action of the acid on the membraneous substances of the fibrous material with the concentrations and limited quantities of acid employed according to the present method is not of the same hydrolysing nature as when high acid concentrations are employed and the hemi-cellulose and cellulose constituents of the vegetable matter more or less quantitatively are hydrolysed to soluble sugars.

If this is not the case it may be supposed that under weak acid conditions a peptisising effect is attained on some of the colloidal substances of the vegetable material, which during the lignification stage of the cell growth are adsorbed from the sap by the initially-formed cellulosic fibre skeleton. As it has also turned out that the acid treatment according to the present method has a more powerful effect in the defibration of younger and not so intensely lignified vegetable parts, this would seam to lend support to the above supposition. However, the invention is not bound down to this supposition.

The chemical effect of the combined acid and high temperature treatment according to this invention is mainly characterized by a slight hydrolysing action on the hemi-cellulose constituents or more specifically the pentosanes of the original materials.

The method according to the invention may preferably be carried out in an ordinary high temperature defibrator for continuous operation according to U. S. Patent No. 2,145,851, equipped with a continuous feeder, a pre-heater, a disc refiner and a discharging device. The feeder may consist of a plunger mechanism or feed-screw, continuously pressing the solid raw material which has been reduced to the form of chips or chaff into a steam-tight plug through a relatively narrow plug-forming pipe into the pre-heater of the defibrator which ordinarily is working under a steam pressure of 100 to 150 pounds per square inch. If the treating liquid is supplied together with the material it is distributed in suitable fashion by the high pressure prevailing in the plug of the material in the feeding apparatus.

The quantity of solution added is not greater than can be completely absorbed by the raw material, which after the treatment does not seem to be wetter than when ordinary wood chips or cut straw is submerged in water or sprayed therewith, whereafter the excess liquid is allowed to drain from the material.

In using the plunger feeder or screw feeder described above any excess of acid solution is pressed out from the raw material. This liquid should be collected and returned for moistening new raw material.

Thus when fed into the closed defibrator system the material is in a substantially dry state.

When the material has passed through the plug-forming pipe it swells when the mechanical pressure is relieved. The steam then flows into the pores of the expanding fibrous material whereby a rapid heating of the material and the treating liquid is achieved.

The material and the chemical treating liquid absorbed therein are afterwards further conveyed to a built-in disc refiner in the closed defibrator system, where the mechanical defibration takes place after the material has attained substantially the temperature of the steam environment.

The finished pulp is thereafter discharged to the outside atmosphere and if required subjected to further treatment such as washing, beating, screening etc., before it is formed to board, paper etc.

Although very good results have been obtained when the method according to the invention is carried out in a defibrating plant according to the design above described other devices may also be employed. Thus, for example, the disc refiner may be replaced by a defibrator of the centrifugal mill type etc.

The chemical solution, e. g. dilute sulphuric acid, may be supplied to the fibrous material in different ways. Thus, the material may be saturated with the chemical solution beforehand or this solution may be sprayed in the form of a fine shower over the fibrous material before it is fed into the defibrator. The solution may also be pumped into the defibrator, whereupon the material is impregnated therewith after having been introduced into the defibrator. The concentration of the solution must be regulated according to the method chosen of supplying it to the fibrous material. It is important that the solution shall be distributed as uniformly as possible in the fibrous material.

When mineral acids are employed, only slight quantities are required in comparison with the quantity of vegetable matter treated. In order to produce a straw pulp capable of being easily ground there is thus required for example 0.5–1% by weight of concentrated sulphuric acid calculated on the basis of the quantity of straw considered as dry, although a marked effect is obtained even with 0.2% of acid. With an addition of 1–3% acid it is possible to produce a stock which directly or after only slight subsequent grinding may be employed for the production of paper or board. From the standpoint of plant operation it may be suitable to add the diluted acid to the straw immediately before the feeding device of the defibrator. The degree of dilution should be so balanced that the natural moisture content of the material to be treated, condensed steam etc. and the added quantity of solution should not be less than 65 gallons per ton of raw material calculated as dry and not exceeding 530 gallons per ton of material, when the material is subjected to the mechanical treatment. The most suitable quantity of total moisture content lies within the range of 130 to 400 gallons per ton of dry material. However, by varying the amount of acid per unit of fibrous material the degree of softening and fibrillation and the beating qualities of the pulp may be varied within wide limits.

The below table given by way of example shows how the freeness of the pulp obtained varies for different additions of acid used when wheat straw is defibrated at a manometric steam pressure of 115 lbs. per sq. in.

Sulphuric acid 0%, freeness 23.1 seconds
Sulphuric acid, 0.2%, freeness 32.0 seconds
Sulphuric acid, 0.5%, freeness 41.0 seconds
Sulphuric acid, 1.0%, freeness 65.5 seconds
Sulphuric acid 1.5%, freeness 110.0 seconds
Sulphuric acid 2.0%, freeness 174.0 seconds
Sulphuric acid 2.5%, freeness 291.0 seconds
Sulphuric acid 3.0%, freeness 435.0 seconds
Sulphuric acid 4.0%, freeness 5400.0 seconds

These results are obtained for a quantity of moisture of approximately 375 gallons per ton of straw calculated as dry. The freeness is measused by determining the dewatering time of 10

litres of a 1.3% pulp suspension on a screen 8 inches in diameter with a manometric vacuum of 40 inches of water.

Thus, when the raw material after the addition of the acid solution has a pH value of 2–3.5 a pulp is obtained which without any further, or in any event after only slight subsequent beating and refining, may be employed for the production of insulating boards, which are to be dried in a roller drier. On the other hand, if high temperature defibration of straw is effected without the use of acid the pulp must be intensively beaten in order that the fibres may acquire adequate felting qualities in order to be employed for the production of such porous fibre boards.

If, for example, in the treatment of straw so much acid is added that a pH-value of 1–2 is reached, a stock will be obtained which has such characteristics that it even without subsequent grinding may be employed for the production of paper and board. In the case of pH-values of less than 1, the defibration must be completed within a very limited time, as otherwise the obtained fibre stock will be inferior or even useless for papermaking purposes. As a matter of fact, the material obtained may then be broken down to such a consistency that it cannot be used as paper stock.

The moisture content of the material present at the mechanical treatment may of course be varied within quite wide limits but should be regulated in order to obtain best results. If too low a moisture content is used it is difficult to distribute the acid solution in the material in a satisfactory way. Furthermore the mechanical defibration also calls for a certain minimum of moisture. The mechanical energy is converted into heat whereby a part of the moisture evaporates preventing a harmful temperature rise of the material in the mechanical defibration zone. Therefore the moisture content should not fall below 65 gallons per ton of fibre material calculated as dry.

On the other hand the moisture content should be limited, since—if much water is present—the acid will be diluted to too great an extent. This weakens the effect of the solution on the raw material. In regulating the moisture content of the material consideration should be taken of the moisture which is condensed in heating the material and the original moisture with steam in the defibrator. Thus the moisture, including the natural moisture of the fibrous material, the added solution and the water together with the condensed steam in the defibrator should not exceed 7–10 lbs. per lb. of fibrous material calculated as dry.

As the chemical effect of a certain amount of chemicals per unit weight of fibrous material is dependent upon the hydrogen ion concentration of the solution, the effect decreases if the solution is diluted. The dilution should therefore be so regulated that the treatment is carried out at a pH-value of the solution of 2–3.5 and in any case confined within the limits of 1–4. If the pH-value lies above 4–5 the desired effect is not attained. The degree of acidity changes during the treatment in such a way that an increase of the pH-value can be established after treatment has been carried out.

Since the method according to the invention is carried out at temperatures of over 212° F. it is obvious that if larger quantities of water are present, the steam consumed for heating purposes will be greater and therefore the quantity of water also on account of this consideration must be limited. In addition, the mechanical defibrating effect is lessened if too much water is present.

If the material is impregnated with the acid solution for 10–20 hours prior to mechanical treatment practically the same results will be obtained as when the impregnation takes place only a short time prior to the material being introduced into the preheater of the defibrator, assuming a uniform saturation. The action of the acid on the material is dependent both on the temperature and on the period of exposure to heat. Therefore, it is only after the fibrous material saturated with the solution is heated in the defibrator that the supporting chemical effect on the mechanical defibration is attained. During the treatment a limited quantity of acid is consumed. If only so much acid is added that it has been totally consumed, the treatment may be extended by some minutes without causing any serious effects, although even in that case consideration must be taken of the decomposing effect of the high temperature on the fibrous material.

If, on the other hand, an excess of acid is present, the time of treatment must be limited, since then a too extensive decomposition of the fibrous material may result. The time of treatment in the defibrator and the degree of acidity in the fibrous material must, therefore, be accurately weighed against one another. The effect can, therefore, within certain limits be regulated by a variation of the time of treatment, that is the time during which the material is exposed to the action of the high temperatures. In normal cases a time of treatment of 20 seconds–2 minutes is sufficient. It should not be extended beyond 2–3 minutes, especially when strong acids are used, such as sulphuric, hydrochloric or nitric acids. If the process according to the invention is carried out with a limited quantity of weak acids or solutions of acid salts the treatment may be extended up to 10 minutes.

Sulphurous acid may also be used according to the method of the present invention. Straw may thus be impregnated with a solution containing SO_2. Upon introducing the impregnated material in the heated steam environment of the closed defibrator system SO_2 gas will evaporate and cause the total pressure to rise above the steam pressure corresponding to that of saturated steam. This should be considered if the temperature in the defibrator is controlled by the aid of the manometric pressure.

The invention also comprises the fibre pulp produced according to the process hereinbefore described.

The fibre pulp produced with the method according to the present invention is characterized by good beating qualities, which makes it suitable for many papermaking purposes although the lignin constituents of the original material are retained. In this respect it differs from pulp produced by ordinary chemical or semi-chemical methods, where the main efforts have been aimed at the dissolving out of the lignin constituents and a retention of the cellulosic part of the original material. The pulp produced according to this invention is therefore not suitable for bleaching with known methods.

The production costs of pulp with the method according to the present invention are low in comparison with, for instance, the production cost of even cheap grades of straw pulp. This

will make it possible to use the pulp for purposes where cheapness is a main consideration, e. g. for certain grades of box board, corrugated liners, etc.

If straw acidified with sulphuric acid is pulped with the method according to the present invention a pulp of greater uniformity is obtained than when ordinary alkaline pulping methods are used. E. g. the nodes ordinarily present in common straw pulp are not present and therefore the screening operation is simplified. The color of the pulp and products produced therefrom is pleasing while this new pulp also in this respect compares favourably with other pulps, produced in the old manners.

What we claim is:

1. The process of producing fibrous pulp from straw comprising wetting the straw with from 0.5 to 3% sulphuric acid based on the dry weight of the straw and in an amount not greater than can be completely absorbed by the straw but sufficient to maintain a pH value of from 1 to 4, subjecting said straw to an atmosphere of steam at 300° F. to 350° F., and at the corresponding pressure for 20 to 180 seconds, and then mechanically defibrating said straw while still in the presence of said acid and said steam atmosphere.

2. The process of producing fibrous pulp from straw comprising impregnating the straw with from 0.5 to 3% mineral acid based on the dry weight of the straw and in an amount not greater than can be completely absorbed by the straw but sufficient to maintain a pH value of from 1 to 4, subjecting said straw to an atmosphere of steam at from 300° F. to 350° F. and at the corresponding pressure for 20 to 180 seconds, then mechanically defibrating said straw while still in the presence of said acid and said steam atmosphere.

3. The process of producing fibrous pulp from straw comprising impregnating the straw with from 0.5 to 3% mineral acid based on the dry weight of the straw and in an amount not greater than can be completely absorbed by the straw but sufficient to maintain a pH value of from 1 to 4, subjecting said straw to an atmosphere of steam at from 270° F. to 380° F. and at the corresponding pressure for 20 to 180 seconds, then mechanically defibrating said straw while still in the presence of said acid and said steam atmosphere.

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