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PRODUCTION OF TEXTILE FIBERS FROM BAST FIBER MATERIAL BY ALKALINE DIGESTION

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My invention relates to the production of textile fibres from bast fibre material, such as flax, hemp, jute, ramie and the like.

The customary method of obtaining spinning fibres from bast fibre material is to subject the crude material to a bacteriological retting process after which the woody material can be easily separated from the fibres by a mechanical treatment. This retting process has the drawback that it takes very much time and therefore many processes have been proposed to replace this retting process by a treatment with alkaline solutions, such as soda lye. In practice these "chemical retting" methods have met with little success, which is mainly due to the fact that the crude 15 material only consists for approximately 1/7 part of spinning fibres so that much lye is necessary to remove the 6/7 parts of nonfibrous woody material.

It has also been proposed to remove the woody 20constituents by subjecting the unretted crude bast fibre material to a mechanical treatment, such as scutching. In this way about % of the woody material may be removed. If one attempts to remove the rest of the woody constituents from 25 this unretted scutched material by subjecting it to a treatment with lye, it appears that after washing out the lye the fibres have a strong tendency to stick together on drying, even in case that the unretted scutched flax is brought $_{30}$ into the form of loose spinnings before the treatment with soda lye.

The object of my invention is to remove this drawback.

According to my invention unretted bast fibrous 35 material, particularly flax straw, is subjected to a mechanical treatment in order to remove the greater part of the woody constituents. A suitable mechanical treatment to this end is the wellknown customary scutching operation. The 40 fibres being deprived in this mechanical way of the larger part of the woody material, are then subjected to a chemical treatment in order to further remove the woody constituents. chemical treatment may consist in subjecting the 45 fibrous material to the action of alkaline solutions. To this end soda lye, e. g. a 10% solution of sodium hydroxide in water, is very suitable, but aqueous solutions of other alkaline substances, e. g. potassium hydroxide, sodium carbonate, and 50 the like, may be used instead. The duration of the treatment depends on the temperature and the strength of the lye. At high temperatures a shorter duration is required than at lower temperatures, e. g. at the boiling point of the lye a 55 solvent the alkaline lye is removed. The greater

treatment during half an hour or three quarters of an hour is generally sufficient, but at 80° C. the treatment requires a few hours and at still lower temperatures a still longer duration is necessary.

The strength of the lye should in general not be lower than 5% and preferably not more than 15%. I prefer an aqueous sodium hydroxide solution of 8-12% and to subject the scutched fibrous material to the action of this boiling lye during 30-45 minutes.

After removal of the lye the moist fibre mass is extracted with an organic solvent in order to prevent that the fibres will stick together on drying. It appeared that by this treatment the small amount of gummy or pectineous substances causing the stickness of the fibres is removed. Though any organic solvent dissolving these substances may be used, it is preferred to employ organic solvents miscible with water. Suitable organic solvents are for instance methanol, acetone, aethanol, propanol or diaceton alcohol. After the extraction treatment with these organic solvents, the solvent remaining in the fibre mass is removed. This may be done by drying, evaporation or the like.

In order to prevent any sticking together of the fibres it is recommended to execute this drying treatment at a low temperature and therefore the use of low boiling organic solvents as extraction agents is preferred. Such low boiling organic extraction agents are e. g. methanol or acetone.

It is also possible, however, to extract the fibrous material by means of higher boiling organic solvents, such as aethanol, propanol or diacetone alcohol and after the mechanical removal of the greater part of these solvents to remove the remaining part thereof by means of lower boiling organic solvents, e. g. acetone. In this case a lower boiling solvent not miscible with water may be used, such as ether.

The removal of the organic solvent from the fibre material after the extraction may be executed by methods known per se, e. g. the organic solvent may be evaporated. To this end a hot current of gas, e. g. air may be circulated through the fibrous material. The solvent is recovered from this gas by condensation. In this way substantially no solvent is lost which makes the process very economical.

If desired the solvent may be evaporated in vacuo. In this case it is not necessary to make use of low boiling solvents, but the danger of solvent losses is greater.

Before the extraction by means of an organic

part may be mechanically eliminated after which the further part may be washed out with water. Any residual sodium hydroxide or other alkaline substance still remaining may be neutralized, e. g. by means of sulfuric or hydrochloric acid.

According to a modification of my invented process the fibrous mass may be subjected to a bleaching treatment after the treatment with alkaline lye. To this end the fibrous mass after the mechanical removal of the excess of alkaline 10 lye, may be subjected to the action of an aqueous solution of an oxidizing agent, e. g. a hypochlorite, hydrogen peroxide or other active oxygen compounds. After this treatment the alsubstance, e. g. hydrochloric, sulfuric or carbonic

By this combination of scutching, treatment with lye, bleaching operation and extraction by means of an organic solvent very light-coloured 20 fine fibres of excellent quality are obtained. The process is highly economical, particularly in case that the lye and the hypochlorite are prepared in the plant by electrolysis.

For instance if the necessary 10% sodium hy- 25 droxide solution is prepared from sodium chloride by electrolysis according to the well-known mercury process, chlorine and hydrochloric acid are formed besides the sodium hydroxide solu-In case that the electric current for the electrolysis is obtained by means of steam the exhaust steam may be utilized for the distillation of the organic solvent and for heating the lye bath.

The hydrochloric acid formed by the elec- 35 trolysis is preferably not used for the neutralization which I prefer to realize by means of sulfuric acid, but for converting the woody constituents, removed from the bast fibres into sugar material, which may be used in fruit preserving or in cattle 40 fodder.

My process is particularly suitable for the production of spinning fibres from flax straw but can also be applied to other bast fibres requiring a "chemical retting."

My invention is further illustrated by means of the following example, but is not restricted

Crude flax is subjected to scutching in the usual way. The scutched flax is completely submerged in 10% soda lye (which may have been prepared by electrolyzing a common salt solution) and heated therein at boiling temperature during half an hour to three quarters of an hour. The excess of lye is removed by a mechanical treatment, e. g. pressing out or centrifugal action and returned to the bath for the treatment of further scutched flax.

The fibrous mass with the lye remaining therein is transferred to a bath with water and chlorine is slowly introduced. In this way sodium hypochlorite is formed which bleaches the fibrous material. After the bleaching action the fibre mass is rinsed in plain water. Any residues of soda lye are neutralized by means of sulfuric or hydrochloric acid.

The excess of water is mechanically removed from the fibre mass, e. g. by passing the mass between rollers under pressure. The damp fibre mass so obtained is extracted by means of acetone 70° in a proportion of six litres of acetone to 1 kg. of moist fibres. This extraction treatment is repeated three times with two litres of acetone each time. The acetone remaining in the fibre

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current of hot air or by vacuum. From this air the acetone may be recovered by condensation and the mixture of acetone and water so obtained can be separated by means of fractional distillation. The acetone so recovered may then be used for the extraction of further flax material.

The dried fibres thus produced are now ready for the further mechanical treatment such as hackling or spinning.

I claim:

1. A process for converting unretted bast fibrous material into dry non-cohesive fibres ready for hackling or spinning, consisting of the steps of: subjecting said unretted fibrous material to kali still present is neutralized by an acid or acid 15 the action of an aqueous alkali solution at a temperature between about 80° C. and the boiling temperature of said alkali solution to remove woody constituents from said bast fibrous material, said alkali solution having a concentration of between about 5% and 15%, removing said alkali solution from the resulting fibers by washing said fibres with water, extracting the water from the resulting moist washed fibres with a water miscible organic solvent selected from the group consisting of methanol, ethanol, propanol, acetone and diacetone alcohol, and removing said solvent from the resulting fibers, to thereby produce dry non-cohesive fibers.

2. A process according to claim 1 wherein said tion. This chlorine is used for the bleaching. 30 alkali solution comprises a compound selected from the group consisting of sodium hydroxide, potassium hydroxide and sodium carbonate.

3. A process according to claim 1 wherein said organic solvent is acetone.

4. A process according to claim 1 wherein said solvent is removed from said fibers by evaporation.

5. A process for converting unretted bast fibrous material into dry non-cohesive cellulose fibers ready for spinning, consisting of the steps of: subjecting said unretted fibrous material to the action of an aqueous alkali solution at a temperature between about 80° C. and the boiling temperature of said alkali solution to remove woody constituents from said bast fibrous material, said alkali solution having a concentration of between about 5% and 15%, bleaching the resulting fibers, washing the resulting bleached fibers with water, and extracting the water from the resulting moist fibers with a water miscible organic solvent selected from the group consisting of methanol, ethanol, propanol, acetone and diacetone alcohol, to thereby produce dry-noncohesive fibers.

6. A process for converting unretted bast fibrous material into dry non-cohesive fibers ready for spinning, consisting of the steps of: subjecting said unretted fibrous material to the action of an aqueous alkali solution at a temperature between about 80° C. and the boiling temperature of said alkali solution to remove woody constituents from said bast fibrous material, said alkali solution having a concentration of between about 5% and 15%, washing the resulting fibers with water, extracting the water from the resulting moist washed fibers with a high boiling water miscible organic solvent selected from the group consisting of methanol, ethanol, propanol, acetone and diacetone alcohol, extracting said organic solvent from said fibers with a lower boiling organic solvent selected from the group consisting of methanol, acetone and ether, and removing said low boiling solvent from said fibers.

7. A process for converting unretted bast fimass is then removed by means of a circulating 75 brous material into dry non-cohesive fibers ready

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for spinning, consisting of the steps of: scutching said bast fibrous material to remove the larger part of the woody material, subjecting the resulting scutched fibers to the action of an aqueous alkali solution at about the boiling temperature of said solution for one half to three quarters of an hour to remove further woody constituents from said bast fibrous material, said alkali solution having a concentration of between about 5% and 15%, said alkali solution comprising a compound selected from the group consisting of sodium hydroxide, potassium hydroxide and sodium carbonate, mechanically removing said alkali solution from said fibers, bleaching said fibers in an aqueous solution containing chlorine, mechanically removing said fibers from said bleaching solution, washing said fibers with water, extracting the water from the resulting moist washed fibers with a water miscible organic solvent selected from the group con- 20 sisting of methanol, ethanol, propanol, acetone and diacetone alcohol, and removing said solvent from said fibers by evaporation, to thereby produce dry non-cohesive fibers.

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References Cited in the file of this patent UNITED STATES PATENTS

	Number	Name	Date		
5	108,487	Keen	Oct.	18,	1870
	962,173	Stark	June	21,	1910
	1,730,387	Schur	Oct.	8,	1929
	1,806,703	Ott	Мау	26,	1931
	1,816,394	Mueller	July	28,	1931
10	1,829,763	Schorger	Nov.	3,	1931
	2,032,123	Freudenberg et al.			
	2,237,295	Akin	Apr.	8,	1941
		FOREIGN PATENTS	3		
15	Number	Country	Date		
	216,892	Germany	Dec.	6,	1909
	300,527	Germany	Sept.	10,	1917
	60,524	Netherlands	Feb.	16,	1948
	82,982	Switzerland	_ Mar	. 21,	1919
20		OTHER REFERENCE	ES		

OTHER REPERENCES

Technical Association Papers, Series 23, pp. 137 and 198 (1940).

Manufacture of Pulp and Paper, 3rd Edition, vol. III, sec. 5, pp. 3-5, and sec. 4, p. 80 (1937), published by McGraw-Hill, New York.