

## UNITED STATES PATENT OFFICE

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## PREPARATION OF STRAW CELLULOSE

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The present invention relates to the treatment of straw and more particularly to a process for the preparation of straw cellulose suitable for manufacturing high grade paper.

When preparing cellulose from wood according to the sulphate process, the wood is digested in an aqueous alkaline solution containing 20% sodium hydroxide and 5% sodium sulphide based on the weight of the dry wood, the quantity of water used being dependent on the type of apparatus and process employed. Generally this quantity of water varies between two and seven times the weight of the wood. The yield of cellulose is in the neighborhood of 50% of the dry wood. However, this same process applied to straw only yields about 30% of the dry straw and the paper manufactured from this cellulose is of poor quality in that it has a low tensile strength and a low number of double folds. (The double fold test is conducted in a machine that bends the paper first in one direction and then in the opposite direction and the number of bends before the paper breaks determines the number of double folds.)

It is also well known to treat straw with acid to effect prehydrolysis before digesting in an alkaline solution. By such a treatment a cellulose with high alpha-cellulose content is then obtained, which is a suitable raw material for the manufacture of rayon, but in using this method for straw, the cellulose yield is decreased even further, i. e., to about 25%, and the cellulose obtained is not particularly suitable for manufacturing paper.

It is possible to prepare a straw cellulose in practical yields and suitable for manufacturing paper by utilizing a two-stage digestion treatment. The first step consists of partially digesting the straw in a weak alkaline solution and completing the treatment by means of gaseous chlorine (Dutch Patent No. 63,978). However, this method is very expensive because it necessitates the use of a large quantity of chlorine, namely, about 20% of the weight of the dry substance to be chlorinated.

It is therefore an object of the present invention to provide a process that is considerably less expensive than the processes heretofore employed.

Another object is to utilize a process in which substantially higher yields of cellulose are obtained from straw, whereas the consumption of

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the chemicals is considerably less than hitherto required.

It is a further object of this invention to produce a high grade cellulose suitable for manufacturing paper which in turn has improved physical and chemical properties.

Other objects and advantages of the invention will be apparent from the following detailed description.

According to this invention, straw is introduced into an aqueous alkaline solution and is digested therein for from 2 to 3 hours, preferably for about 2 hours and 15 minutes. The temperature of the solution is maintained between 115° C. and 130° C., preferably at about 120° C. The total alkaline agents employed are not less than 7% and not more than 9% based on the weight of the dry straw and consist of about 80% sodium hydroxide and about 20% sodium sulphide. These percentage ranges can be satisfied if the sodium hydroxide varies from 5.6% to 7.2% while the sodium sulphide varies from 1.4% to 1.8%. Apparently the optimum percentages of the alkaline agents based on the weight of the dry straw are 6.4% sodium hydroxide and 1.6% sodium sulphide.

It was determined that the best results were achieved when the digesting operation took place using a straw-liquid ratio of 1:2. The straw-liquid ratio is the ratio of the dry weight of the straw (in kg.) to the volume of the liquid (in liters). Since the dry straw retains a certain amount of moisture from the atmosphere, the moisture content of air-dry straw must be taken into consideration. Thus, if the air-dry straw contains 10% of moisture and 100 kg. are used, in order to provide an aqueous alkaline solution of 6.4% sodium hydroxide and 1.6% sodium sulphide, it is necessary to use  $90 \times 0.064$  or 5.76 kg. sodium hydroxide and  $90 \times 0.016$  or 1.44 kg. sodium sulphide. These alkaline agents are dissolved in a total liquid volume of  $(2 \times 90) - 10$  or 170 liters of water.

In reaching the conclusions that about 120° C. is the most desirable temperature for the digesting operation, and that about 6.4% sodium hydroxide and about 1.6% sodium sulphide, based on the weight of the dry straw, are the most satisfactory concentrations of alkali in the solution, several experiments were conducted varying these conditions. At this particular temperature and these particular concentrations, it was

determined that the highest yield of cellulose was obtained while the subsequent removal of the lignin was effected with the least amount of chlorine. Moreover, the properties of the paper manufactured from the straw cellulose were excellent.

In the first series of experiments, the straw was digested in an aqueous alkaline solution maintained at different temperatures. The straw-liquid ratio was 1:2, and the alkaline solution contained 6.4% sodium hydroxide and 1.6% sodium sulphide, based on the weight of the dry straw. In each experiment the air-dry straw contained 10% moisture and the digesting operation was conducted for two and one-quarter hours. Table I given below confirms the fact that the highest yield of cellulose and the lowest consumption of chlorine was obtained at 120° C.

Table I

Temperature	Cellulose yield in percent calculated on dry straw	Chlorine consumption in percent calculated on dry cellulose
160° C	40	22
140° C	44	20
130° C	44	15
120° C	47	11
110° C	40	12

The following table involving the same series of experiments shows that the properties of the paper manufactured from the straw cellulose were extremely better when the digesting operation took place at 120° C. as compared with the operation in which the digesting took place at 160° C. The breaking length in km. refers to the length of paper that breaks under its own weight when freely suspended from a support.

Table II

Temperature	Breaking length in km.	Number of double-folds
160° C	6.3	200
140° C	7.2	600
130° C	7.6	1,000
120° C	8.0	1,200
110° C	8.5	1,500

Additional experiments showed that when digesting at 120° C., decreasing the alkali content did not give an improvement in the cellulose yield and had the disadvantage that the chlorine consumption was greater. The results of these experiments are tabulated as follows:

Table III

Temperature	Percent NaOH+Na <sub>2</sub> S	Chlorine consumption in percent calculated on dry cellulose	Cellulose yield in percent calculated on dry straw
120° C	6.4+1.6	11.5	47.0
120° C	6.0+1.5	13.0	46.0
120° C	5.6+1.4	16.7	43.0

Increasing the sodium hydroxide and sodium sulphide content gave no better results insofar as the cellulose yield was concerned, even when the temperature was decreased. This is clearly shown from the following table.

Table IV

Temperature	Percent NaOH+percent Na <sub>2</sub> S	Cellulose yield in percent calculated on dry straw
150° C	12+3	43.9
140° C	12+3	45.0
130° C	12+3	45.9
120° C	12+3	40.6

It can be seen from the above tables that the best all around results are achieved when using the amounts of alkali and temperature mentioned in the first series of experiments. Therefore, the processes of the invention are considerably cheaper with respect to the chemicals used, including the chlorine consumption, which materially reduces the expense of the operation, and in addition the process gives a higher yield of cellulose which is particularly well suited for the manufacture of high grade paper.

What is claimed is:

1. A process for the preparation of straw cellulose suitable for manufacturing paper which comprises digesting the straw for a period of time not more than about 3 hours and not less than about 2 hours in an aqueous solution consisting essentially of sodium hydroxide and sodium sulphide maintained in a temperature range between 115° C. and 130° C. and having a total content of alkaline agents from 7% to 9% based on the weight of the dry straw, said alkaline agents consisting of about 80% sodium hydroxide and about 20% sodium sulphide, the straw-liquid ratio being about 1:2.

2. A process for the preparation of straw cellulose suitable for manufacturing paper which comprises digesting the straw for a period of time not more than about 3 hours and not less than about 2 hours in an aqueous alkaline solution maintained at a temperature of about 120° C. and consisting of about 6.4% sodium hydroxide and about 1.6% sodium sulphide, each based on the weight of the dry straw, the straw-liquid ratio being about 1:2.

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