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PROCESSES FOR THE EXTRACTION OF CELLULOSIC FIBRES FROM VEGETABLE MATERIAL

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This application is a continuation-in-part of my application Serial Number 153,993 filed July 16, 1937.

The invention is for improvements in or relating to processes for the extraction of cellulosic fibres from vegetable material and particularly from vegetable material such as the stalks of wheat, barley, oats, rice and other cereals, and of flax, maize, cotton, hemp, and from material such as sisal, phormium tenax, bagasse, different grasses such as esparto, tow-residues and residues from the manufacture of textile materials. One of the objects of the invention is to produce cellulosic fibres suitable for the manufacture of high-grade and other forms of paper and cardboard and the like. The cellulosic fibres which are present in the materials set out above are for the most part embedded in ligneous or woody matter encrusted with waxes, resins, pectic, mucic and albumenous matters and mineral compounds (e. g. silica compounds). It is already known how to extract the cellulosic fibres by dissolving out the above mentioned substances by treating the vegetable material with alkali and chlorine. The alkali removes a substantial part of the encrusting substances, while the chlorine reacts with the ligneous or woody matter. The concentration of the alkali solution usually employed has been comparatively high, amounting to 10% or higher, and similarly, where chlorine has been used, it has been employed in an undiluted state. Paper manufactured from the resulting fibres has tended to be harsh and crackly.

The temperature under which the alkali reaction of the above treatment is carried out affects the physical characteristics of the resulting fibre. High temperature conditions tend to damage the fibre, for example the fibre is increasingly damaged with an increase of temperature over 150° C. The reactions are exothermic and high temperatures are liable to be built up.

Since the reactions are exothermic, the temperatures built up are dependent inter alia on the velocity at which the reactions take place and the rate at which the heat evolved is dissipated through the mass of the material and through the walls of the reaction vessels. The velocity at which the reaction takes place in the alkali treatment is dependent on the amount of alkali employed for a given weight of material treated and the strength of the alkali solution.

In the chlorination treatment, the reaction is also exothermic. Cellulose is also adversely affected by any oxidation or bleaching action during the chlorination treatment as has occurred in the processes heretofore used. In my process the quantities of reagents used and the time that they are in contact with the material treated are so regulated that high temperatures are not developed and during the chlorine treatment the non-cellulosic materials are removed by virtue of a chemical reaction in which chloro-lignins are formed. There is little harmful bleaching or oxidizing action.

My process, briefly described, involves treating the material from which the cellulose is to be extracted with an alkali solution of a certain strength and quantity, drying the material, treating the dried material with chlorine in certain quantities, and for certain periods of time, washing the material after the chlorine treatment and treating the washed material with a second alkali solution. My process may be carried out in apparatus such as is described and claimed in my application Serial Number 153,993, of which the present application is a continuation-in-part. Preferably, the apparatus includes a tower into the top of which the raw material from which the cellulose is to be extracted may be continuously fed. The tower has pipes and sprays by which the alkali solution can be thoroughly mixed with the vegetable material being treated. The material moves by gravity to the bottom of the tower from which it is withdrawn and led to a press where the moisture is extracted from the material. The apparatus also preferably includes a tower in which the chlorine treatment is carried on. After the material is treated with alkali and dried, it is conveyed to the top of the chlorine tower down which it then passes under the influence of gravity. The chlorine tower has pipes whereby chlorine may be brought into contact with the material as it passes down the tower. Mixing containers are also provided for washing the material after the chlorine treatment and subjecting the material to a final alkali treatment.

The stalks of plants such as are mentioned at the beginning of the specification contain about 8% lignin by weight of the material and about 42% of waxes, rosins, pectic, mucic and albumenous matter, etc. The quantities of alkali and chlorine to be used in order to keep the temperatures of the reactions at the proper levels vary with the material being treated. I have also found that in order to insure the formation of chloro-lignins during the chlorine reaction the strength and quantity of the alkali used during the treatment of the vegetable material with alkali should be just sufficient to remove approximately 30% of the non-cellulosic content of the vegetable material. If approximately 30% of these materials are removed during the alkali treatment, a sufficient quantity of the non-cellulosic materials remains in the vegetable material during the chlorination treatment to insure the formation of chloro-lignins.

For the first alkali treatment, I employ a caustic soda solution of 1 to 2% strength. When such a solution is employed the relative proportions of the reacting substances are selected to be one part by weight of vegetable material to four to six parts of the solution. The reaction is carried out in an open vessel without pressure for a period from one to four hours according to the nature of the material and the strength of the solution, the extent of the initial disintegration of the material and the temperature may vary from 80° C. to 100° C. The quantity of alkali used is varied in accordance with the material being treated so that only approximately 30% of the non-cellulosic material is removed by this first alkali treatment. In terms of weights of materials, alkali in an amount approximately 1/16 the weight of the vegetable material should be used. The balance of the non-cellulosic material is carried over with the material and is used during the chlorination treatment to form hydrochloric acid. The presence of hydrochloric acid in an ionized state (as it is during the chlorination treatment) reduces the formation of hydrochlorous acid which, if formed, would dissassociate to release oxygen. This oxygen would have an oxidizing or bleaching effect on the cellulose. Oxidation impairs the quality of the cellulose. Hydrochloric acid is also harmful to cellulose but the reaction between the hydrochloric acid and cellulose is much slower than oxidation. In my process the cellulose is not permitted to remain in contact with the hydrochloric acid for so long a time that the hydrochloric acid materially affects the cellulose.

After the alkali treatment the vegetable material is washed with water and dried as by passing it through a screw press. Preferably the material is broken up and converted into a floccular state by passing it through spiked rollers and then raised by air blowers to the top of the chlorination tower.

After this drying process the material has a dryness factor of from 25 to 40. Before the chlorination treatment the material should be relatively dry so that there is not an excess of water present during the chlorination treatment. An excess of water will dilute the hydrochloric acid, thereby lowering the hydrogen-ion concentration and permitting the formation of hydrochlorous acid which, as stated above, oxidizes the cellulose.

The chlorination treatment is effected by causing chlorine gas diluted with air to pass through the vegetable material. Preferably the chlorine gas and reacting mass are both moist with water. The function of the air is twofold. It reduces the velocity at which the reaction takes place and thus the rate at which heat is evolved, and it also forms a vehicle for conducting away the heat. The proportion of air used with the chlorine is dependent on the nature of the vegetable material being treated and it may vary from .5 to 1. parts by volume of air to one part by volume of chlorine, while the moisture content of the chlorine may be up to its saturation point and the moisture content of the mass may be two to five times the dry weight of the mass itself.

The rate at which the chlorine-air mixture is passed through the vegetable material also varies with the nature of the material being treated, since certain materials can be treated at higher temperatures than others without adversely affecting the cellulosic fibres. For example, wheat straw may be treated up to a temperature of 55° C. while hemp straw may be treated up to 65° C. The rate at which chlorine may be passed through the material per pound of the material per hour varies from 1/16 of a pound to 1/8 of a pound. The length of the chlorination treatment may vary from half an hour to three hours.

The vegetable material passes down through the chlorination tower under the influence of gravity and then into a tank where the material is rinsed in cold water. The material is then passed through a screw press and finally into a mixing vat containing a solution of caustic soda which is weaker than the solution used during the first alkali treatment. However, the solution should have sufficient strength to dissolve the chloro-lignins formed during the chlorination treatment and to remove any non-cellulosic material remaining in the vegetable material being treated.

The regulation of the quantity of the reagents used so as to insure the formation of chloro-lignins during the chlorination step, the attenuated nature of the treatment in a number of steps, the comparatively slow velocity of the reactions and the comparatively low temperatures under which the reactions are carried out result in a high yield of fibrous cellulose of good quality suitable for the manufacture of a high grade of paper having strength, softness and pliability.

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