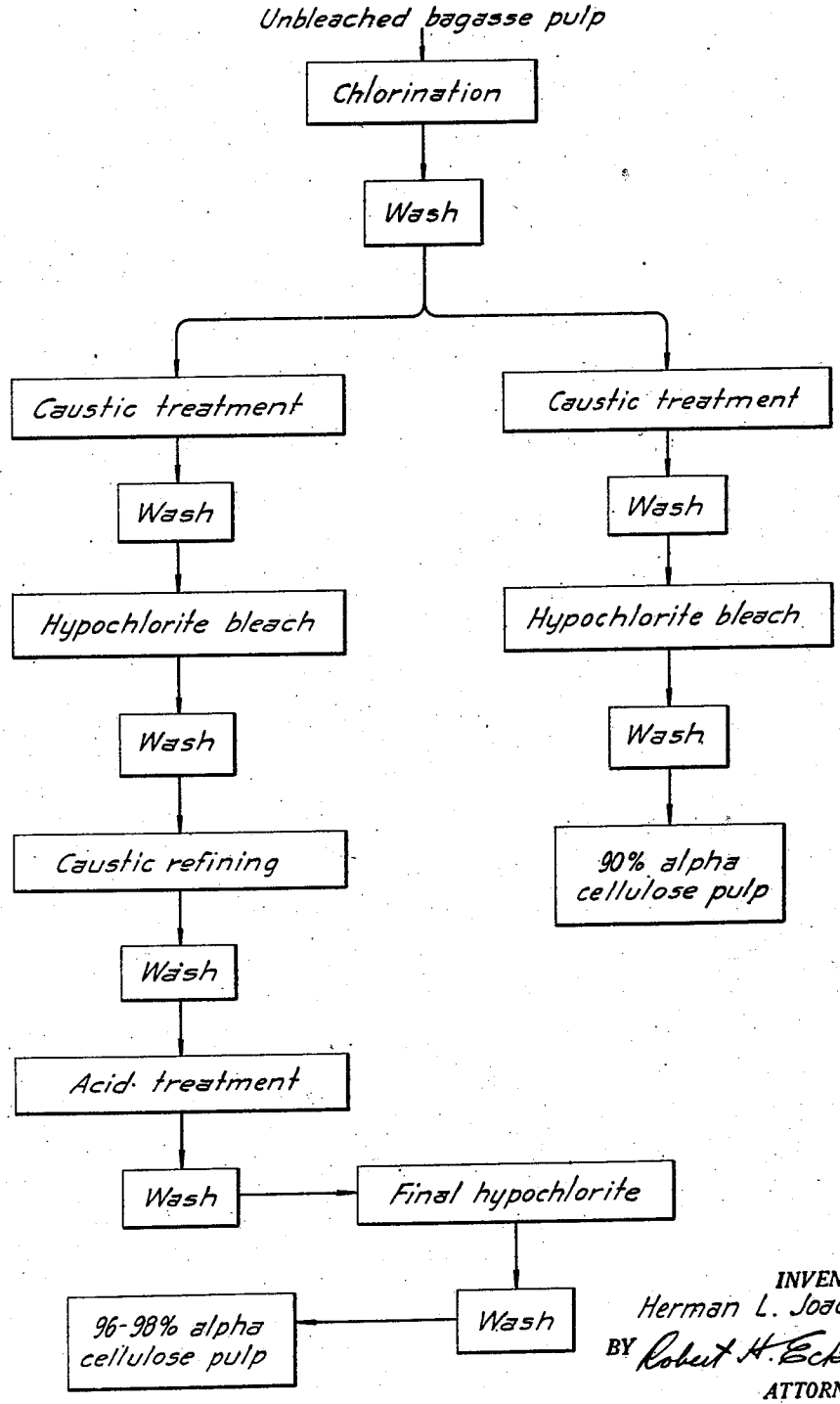
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BLEACHING PULP

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BLEACHING PULP

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6 Claims. (Cl. 8—105)

This invention relates to the chemical refining and purification of a bagasse pulp and to the production of a new alpha cellulose pulp containing 96% and more alpha cellulose, having a low soda solubility, low copper number, a negligible ether solubility and a materially higher reactivity as compared to the reactivity of cotton linters and super-refined wood pulps.

With a constantly increasing demand for alpha cellulose by the film, rayon, explosives and plastics industries, and with the inherent limitation on alpha cellulose pulps derived from wood, namely an alpha cellulose content of 95% and less, a copper number of 0.8 to 1.0, a soda solubility of 3%–3.5% and an ether solubility of from 0.2%–0.5%, attention has been given to the production of alpha cellulose from other cellulosic materials.

While bagasse is one of the most important cellulose bearing crops, because sugar cane from which it is obtained can be rapidly grown, utilization of bagasse as an alpha cellulose source has heretofore not been economically practical. While the literature contains various statements to the effect that bagasse pulp can be treated according to methods suitable to wood pulp, I have found that in fact this was not so. Presumably the difficulties in treating bagasse pulp according to the methods heretofore employed upon wood pulp have largely failed because of the complexity of the bagasse fiber bundles and because of the difference which characterizes bagasse from wood.

I have found that a bagasse pulp, after suitable delignification operation, such as that disclosed in my copending application with John P. Foster, Serial No. 164,212, filed September 16, 1937, can be suitably treated to produce a pulp of high alpha cellulose content, low copper number, satisfactory viscosity, low soda solubility, and negligible ether solubility.

I have further found that when care is taken not to degrade the pulp during refining and purification, a pulp is produced of high alpha cellulose content, 96% and higher. And, further, what is even more important, the pulp has such a physical fibrous structure that it adsorbs water and aqueous solutions, swells and reacts with those reagents commonly employed in the acetate, nitrate, cuprammonium and viscose process far more rapidly and completely than do the alpha cellulose materials known heretofore such as cotton linters and super-refined wood pulp.

Depending on the degree of delignification effected in the pulping operation, a bagasse pulp, as produced from the sugar cane grown in the Hawaiian Islands will require 3½% to 5% by weight on a dry basis of total chlorine to remove impurities present. I have found that initially the pulp, while in aqueous suspension, should be subject to chlorination to only a limited degree. For example, producing high alpha cellulose pulps, 96% and higher, I have found that on Hawaiian bagasse only from 62.5% to 67.5% of the total chlorine demand should be initially supplied to ensure a high yield of pulp of good quality. When it is desired to produce a pulp containing only 90% alpha cellulose, this treatment can be made more drastic, and as much as 75% of the initial chlorine demand supplied in the first of a plurality of chlorinating stages. An excessive increase in the initial chlorine supplied degrades the pulp, lowering the yield of alpha cellulose, lowering the viscosity and increasing the copper number. While the values given are relatively critical for the particular bagasse, and will be critical for other like bagasses, sugar canes grown in other localities differ and these percentages may accordingly vary.

After the initial chlorination operation, I have found that the pulp should be separated from the initial chlorination liquor, washed and then treated with a caustic solution. Thereafter substantially all the remaining chlorine demand is supplied, preferably as a hypochlorite bleach liquor.

When an alpha cellulose pulp of high alpha content is desired, 96% and higher, the pulp is further treated with strong caustic solution, washed and treated with a mineral acid to adjust the viscosity of the pulp. Finally, the pulp is bleached with a very light hypochlorite bleach.

The new 96% and higher alpha cellulose pulp of this invention is particularly characterized in having some properties comparable to those of a high grade of cotton linters, as will be apparent from a consideration of the properties appearing in the following table.

Property	Cotton linters	Bagasse pulp	Super refined wood pulp
Alpha cellulose content	98%	96 -98%	94 -95%
Copper number	0.15- 0.25	0.2 - 0.22	0.8- 1.0
Soda solubility	2 - 3.5%	2 - 2.5%	3.0- 3.25%
Ether solubility	0.15%	0.03- 0.05%	0.2- 0.5%

The new high alpha cellulose pulp of this invention is further characterized in having such a physical fibrous structure that the pulp adsorbs water and aqueous solutions, swells and reacts with the reagents usually employed in the acetate, nitrate, cuprammonium and viscose processes. Comparing the speed of reaction in any one of these operations with the speed when cotton linters or super-refined wood pulp are employed (these materials being chosen for comparison because they represent the best materials known heretofore), it can be stated that

the 96% or higher alpha cellulose pulp of this invention is far more reactive.

The practice of the present invention will be further apparent upon a consideration of the following, wherein, by way of example and not by way of limitation, I have set forth the procedure followed in producing a 96% alpha cellulose pulp from an unbleached bagasse pulp from an Hawaiian sugar cane.

The drawing accompanying and forming a part hereof is in the nature of a flow sheet illustrating the various steps practiced.

The unbleached bagasse pulp had an initial alpha cellulose content of 86% and a copper number of 1.0. It was produced according to the process of the aforementioned copending application.

The pulp was placed in an aqueous suspension of 3–3.5% consistency and chlorinated by the addition of 65% of its total chlorine demand. The chlorination was permitted to continue until only a trace of chlorine could be found. Usually from 20 to 30 minutes were required. During the chlorination the temperature was held at about room temperature, and was not permitted to exceed 80° F. It was found that if the temperature exceeded 80° F. the pulp was degraded. It was also found that the chlorination could be carried on with from 62.5%–67.5% of the total chlorine demand if a high cellulose pulp, 96% and higher, was desired from this bagasse pulp, although bagasse pulps from other types of cane may vary as to the initial chlorine supplied. The thing to avoid was over-chlorination. Some bagasse pulps, by reason of differences in their inherent properties traceable to the original cane and the regions wherein it was grown, could stand more chlorine than others without sacrificing quality and yield.

After the chlorination had continued until only a trace of chlorine was left, the pulp was washed. I have successfully employed a continuous vacuum washer, utilizing warm water and washing until the pH of the wash water had risen to pH 6.5–6.8. Thereafter the pulp was diffused with an alkaline water. The water and pulp were heated to 120°–124° F., from 45 to 60 minutes being required. The pulp, in the aqueous suspension, was held at this temperature for about an hour. The consistency of the pulp in this step was from 5% to 6%, while initially about 2½% of caustic soda on the dry weight of the pulp was added. More or less caustic soda could be employed, but this, it was found, would serve only to decrease or increase the time of treatment. After the diffusion treatment, the pulp was again washed on a vacuum washer with warm water until the pH had fallen to pH 8.2–8.6.

The operations previously recited were carried on continuously or as batch operations, as desired.

The pulp was next treated with a hypochlorite solution, preferably a sodium hypochlorite bleach, although calcium hypochlorite can be employed. Substantially all the chlorine required to remove impurities was added here, and I preferably employed about 25% of the total chlorine demand. The pH of the hypochlorite solution was held initially on the acid side and within the limit of carbonic acid so that, in effect, the operation was carried on with hypochlorous acid. The pulp consistency was about 8%, but as high as 16% consistency could be employed. To facilitate control this operation was usually handled as a batch operation.

After the pulp had been subjected to the action of the hypochlorite for about 45 minutes, caustic soda was added to alkalize the bleach liquor and solubilize any chlorination products. If the caustic was not added, the final pulp was not as bright. I have successfully added a 1% caustic soda solution to alkalize the pulp suspension, although more or less could be used. After the addition of the caustic soda, about 45 minutes were permitted to elapse, after which the hypochlorite treated pulp was washed on a vacuum washer with warm water.

The pulp was then refined and to this end it was subjected to the action of a strong caustic solution. I preferably employed one containing from 1.4 to 1.8 parts of caustic for each part of pulp on a dry basis. This refining treatment was continued for about 2 hours at an elevated temperature, preferably in the neighborhood of 204° F., or between 202° F. and 206° F. This served to dissolve impurities present and raise the alpha cellulose content to the neighborhood of 96%.

When 98% alpha cellulose was desired, the strength of the caustic solution employed was increased toward 1.8 and I have successfully employed a ratio of 1 part of pulp to 1.75 parts of caustic; in producing the 96% cellulose a ratio of 1 part of pulp to 1.64 parts of caustic was employed. The consistency of the pulp during refining could be varied, but I have successfully used about 4% consistency. At the end of the refining operation it was found that the copper number had dropped to about 0.2. After the caustic treatment, the pulp was washed until the pH of the wash water had dropped to about 9.5–10.

The refined pulp was treated to adjust its viscosity. This treatment was carried on at about room temperature for 20 to 30 minutes, with a suitable concentration of a mineral acid such as hydrochloric acid, the pulp being at about 4% consistency. I have found the temperature during the acid treatment should not exceed 80° F. Above this temperature the pulp was ruined rapidly.

Depending on the desired viscosity of the pulp, more or less acid was employed. Other acids as sulphuric, nitric and phosphoric could be used. In one case I employed 7 liters of 36% HCl to 200 pounds of dry pulp. This served to give a pulp of a viscosity of 25 to 38 centipoises in a 1% solution. The acid treated pulp was then washed on a continuous vacuum washer until the pH of the wash water had dropped to about pH 7.2–7.6.

Because the treatment with a strong caustic tended to give a grayish cast, which the acid treatment removed almost but not entirely, I preferably concluded the purification with a final bleach with a hypochlorite solution. This bleach was almost instantaneous, removing whatever gray cast was present in the pulp almost instantly. The remaining chlorine required to make up the initial total chlorine demand was added here as a hypochlorite, either sodium or calcium. The quantity was usually 10% or less of that required initially. The temperature was kept below 80° F. and usually at about room temperature. The bleach was usually very rapid, and 20 to 30 minutes usually sufficed. Thereafter the pulp was washed until the pH of the wash water was about 7.4–7.8. The pulp was then ready for thickening and drying. The pulp produced according to this process was particularly characterized by its high alpha cellulose content,

its low copper number, its satisfactory viscosity, its low soda and ether solubility, and its high reactivity as compared to cotton linters and super-refined wood plups.

In those substances when it was my desire to produce a pulp in which the alpha cellulose content was not so high, about 90%, for competition with lower grades of alpha cellulose materials, the initial chlorine supplied was increased, and I have used as much as 75%. This chlorine was left in contact with the pulp until only a trace remained when the pulp was washed. After washing, the pulp was subjected to a caustic solution containing about 1 part of pulp to 1.15 parts caustic by weight. Holding the pulp in this solution and treating it for about 2 hours at 204° F. served to increase the alpha cellulose content of the resulting pulp to about 90%. A strongly alkaline solution could not be used in this refining operation because the highly chlorinated pulp was quite sensitive to it. Thereafter the material was washed and, depending on the viscosity, was treated with a mineral acid as hydrochloric acid. The acid step was omitted when the viscosity was satisfactory. Thereafter the final chlorine demand was supplied, as sodium hypochlorite, this operation being carried out below 80° F. and usually at about room temperature. The pulp was finally washed.

It is to be understood that conventional equipment can be employed in carrying on the foregoing procedures.

I claim:

1. A process for purification of a substantially delignified but substantially otherwise untreated bagasse pulp to produce a high grade alpha-cellulose pulp comprising treating an aqueous suspension of said pulp with free chlorine in an amount from about 62.5% to about 67.5% of the total chlorine demand of said pulp and at a temperature below about 80° F., washing the chlorine treated pulp, treating the washed pulp with an alkaline liquor, washing the alkali treated pulp, treating the washed pulp with an acid hypochlorite solution containing substantially all additional chlorine required to oxidize remaining impurities, alkalizing said pulp during said hypochlorite bleach, washing the alkalized pulp, treating the pulp with a strong caustic solution, and washing the pulp.

2. A process for purification of a substantially delignified but substantially otherwise untreated bagasse pulp to produce a high grade alpha-cellulose pulp comprising treating an aqueous suspension of said pulp with free chlorine in an amount from about 62.5% to about 67.5% of the total chlorine demand of said pulp and a temperature below about 80° F., washing the chlorine treated pulp, treating the washed pulp with an alkaline liquor, washing the alkali treated pulp, treating the washed pulp with a hypochlorite solution containing substantially all additional chlorine required to oxidize remaining impurities, alkalizing said pulp during said hypochlorite bleach, washing the alkalized pulp, treating the pulp further with a strong caustic solution containing from about 1.4 to about 1.8 parts of caustic for each part of pulp on a dry basis, and washing the pulp.

3. A process for purification of a substantially delignified but substantially otherwise untreated bagassee pulp to produce a high grade alpha-cellulose pulp comprising treating an aqueous suspension of said pulp with free chlorine in an amount from about 62.5% to about 67.5% of the total chlorine demand of said pulp and at a temperature below about 80° F., washing the chlorine treated pulp, treating the washed pulp with an alkaline liquor containing about 2½% caustic soda on the dry weight of the pulp and at a temperature of about 120° F. to render said pulp alkaline, washing the alkali treated pulp, treating the washed purified pulp with a hypochlorite solution containing substantially all additional chlorine required to oxidize remaining impurities, treating the hypochlorite treated pulp with an alkaline solution, washing the alkalized pulp, treating the pulp for about two hours at about 204° F. with a strong caustic solution containing from about 1.4 to about 1.8 parts of caustic for each part of pulp on a dry basis, and washing the pulp.

4. A process for purification of a substantially delignified but substantially otherwise untreated bagasse pulp to produce a high grade alpha-cellulose pulp comprising treating an aqueous suspension of said pulp with free chlorine in an amount from about 62.5% to about 67.5% of the total chlorine demand of said pulp and at a temperature below about 80° F., washing the chlorine treated pulp, treating the washed pulp with an alkaline liquor, washing the alkali treated pulp, treating the washed purified pulp with a hypochlorite solution containing substantially all additional chlorine required to oxidize remaining impurities, treating said pulp with an alkaline solution to render said pulp alkaline, washing the alkalized pulp, treating the pulp with a strong caustic solution containing from about 1.4 to about1.8 parts of caustic for each part of pulp on a dry basis, washing the pulp with a dilute acid at a temperature below about 80° F. to acidify the pulp, and washing the pulp with water.

5. A process for purification of a substantially delignified but substantially otherwise untreated bagasse pulp to produce a high grade alpha-cellulose pulp comprising treating an aqueous suspension of said pulp with free chlorine in an amount from about 62.5% to about 67.5% of the total chlorine demand of said pulp and at a temperature below about 80° F., treating the pulp with an alkaline solution, washing the alkali treated pulp, treating the washed pulp with a hypochlorous acid solution containing substantially all additional chlorine required to oxidize remaining impurities, treating the pulp with an alkaline solution to render the pulp alkaline, washing the alkalized pulp, treating the pulp further with a strong caustic solution, and washing the pulp.

6. A process for purification of a substantially delignified but substantially otherwise untreated bagasse pulp to produce a high grade alpha-cellulose pulp comprising treating an aqueous suspension of said pulp with free chlorine in an amount of from about 62.5% to about 67.5% of the total chlorine demand of said pulp and at a temperature below about 80° F., washing the chlorine treated pulp, and thereafter treating the pulp alternately with caustic solutions and acid chlorine solutions and washing the pulp after each treatment.

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