

UNITED STATES PATENT OFFICE

2,127,765

BLEACHING PULP

Ferri Casciani, Niagara Falls, N. Y., assignor to
Niagara Alkali Company, Niagara Falls, N. Y., a
corporation of New York

No Drawing. Application July 23, 1937,
Serial No. 155,341

3 Claims. (Cl. 8-105)

This invention or discovery relates to bleaching pulp; and it comprises a method of bleaching pulp fiber, especially kraft pulps, wherein the fiber in aqueous suspension is treated with elemental chlorine in amounts somewhat less than is readily taken up, the acid liquor carrying soluble matters is removed and the fiber washed, the washed fiber is again converted into a thin aqueous suspension, is again treated with additional chlorine, this time in amount somewhat more than is readily taken up and the mixture made alkaline, drained, washed and subjected to a mild hypochlorite bleach, the pulp sometimes also being washed after the second chlorine addition and prior to alkalizing; all as more fully hereinafter set forth and as claimed.

Kraft wood-pulps are desirable in certain paper manufactures because of their long fiber length and excellent fiber quality, which makes for high strength, high fold test, tearing test and other strength characteristics in paper, card, etc., produced therefrom. However, they have the disadvantage of having a brownish coloration which makes kraft paper unsightly and unsuitable for many purposes. The stronger pulps are often quite dark.

These pulps can be bleached to a fairly light shade by ordinary methods but bleaching to this extent is difficult, requiring large quantities of reagents and long treatment times. Moreover, kraft pulps always lose some strength in being bleached.

The processes proposed for bleaching kraft pulp generally depart more or less from the old-time practice of using bleach liquor (an alkaline hypochlorite solution) alone. The use of elemental chlorine introduced at one point or another has become common. In one typical method, pulp in aqueous suspension is subjected to the action of chlorine, gaseous chlorine being introduced in large quantities. The chlorine attacks the non-cellulose part of the pulp ("lignin") more or less preferentially, producing soluble products and giving a highly acid, impure liquor. The chlorinated suspension is then treated with enough alkali to neutralize it and to form hypochlorite. The amount of alkali added is ordinarily in sufficient excess to secure considerable alkalinity. Finally, the pulp is subjected to a mild bleaching with an ordinary alkaline hypochlorite; calcium hypochlorite or sodium hypochlorite, as the case may be. The bleaching process always ends with an alkaline bleach of the old time type. In processes of this sort the consumption of chlorine and of alkali is considerable; sometimes sev-

eral hundred pounds per ton of pulp. And the drastic chlorination lessens the strength of the pulp fibers to an undesirable degree. As stated, as a rule, the lighter colored the pulp the less the strength and vice versa, but this is not necessarily true.

There is a need in the art for methods of bleaching kraft pulp which are more economical in chlorine and alkali consumption; and for methods which will bleach without so much degradation of fiber strength.

According to the present invention, there is provided a process for bleaching kraft pulp, using chlorine and alkali, which makes possible a substantial reduction in the consumption of reagents, and which moreover yields a higher strength pulp for the same degree of bleaching than processes hitherto known.

I have discovered that if the pulp in aqueous suspension be preliminarily treated with elemental chlorine in fairly small proportions, less than the amount readily taken up, there is an attack on the non-cellulose matter, putting much of it in soluble form. There is also a considerable development of acidity, the acidity holding some of these dissolved matters in solution. This acid liquor is capable of taking up more chlorine but chlorinating it is, to a certain extent, wasting chlorine.

If this acid liquor be displaced, the pulp fiber thoroughly washed with water and again put into aqueous suspension, on introduction of another portion of chlorine, a smoother action is secured. The second suspension, like the first, is thin enough to pump, pipe and handle, enough water being used to secure this result and produce a suspension in which the small amount of secondary chlorine can be uniformly incorporated. This small amount of chlorine, however, is somewhat more than can be readily taken up and the excess forms hypochlorite on addition of alkali.

The total chlorine required in the two-stage operation described is less than that required in one-stage operation. It appears evident, as stated, that some of the matters preliminarily going into solution and removed in the washing operation of the present invention are capable of taking up a considerable amount of chlorine uselessly.

Another advantage in getting rid of the liquor formed in the first chlorination is that acid is removed. The use of alkali to neutralize this liquor is wasteful and moreover alkalization throws some of the solubles out of solution. The

total requirements for alkali in the present process are considerably less.

Operating in the way described, the total chlorine required for a given amount of bleaching is less and less alkali is needed and there is less action on the cellulose part of the fiber, giving a light color with less loss of strength. The action of the chlorine in the present process is more sharply selective than in conventional processes and a whiter and stronger pulp is produced. The bleached pulp more nearly approaches pure cellulose.

After the two chlorinations I ordinarily add alkali, either caustic soda or lime, in amount sufficient to make the mixture alkaline and to provide hypochlorite bleaching. The pulp is then washed and usually subjected to a final mild bleach with a calcium hypochlorite liquor. I often drain and wash the fiber after the second chlorination and then re-convert it into a suspension prior to addition of the alkali. This washing removes some acid and coloring matters and reduces the alkali required for neutralization, by an amount equivalent to the amount of acid removed.

Considering the process in more detail, in the first step a batch of kraft pulp in an aqueous suspension of ordinary strength (say 3 or 4 per cent) is agitated and chlorine gas introduced, in amount much less than that required for complete reaction upon the non-cellulose constituents; an amount determined by a separate experiment. A suitable length of time is allowed for reaction; to exhaust the available chlorine. The extent of the bleaching secured at this stage is not material. At the end of this time the liquor is removed and the fiber washed with water. For greatest economy, the washing should be quite thorough. The liquor withdrawn and the rinsings are strongly acid with HCl and contain substantial amounts of organic material, varying from 2 to 4 per cent based on the dry weight of the unbleached pulp. The quantity of acid drained off and washed out comes to 2 to 4 per cent (based on the dry weight of the pulp) in most cases.

In the next step the washed fiber is again put into aqueous suspension. As before, it is convenient to have this suspension carry about 3 or 4 per cent of fiber. The aqueous suspension is again treated with chlorine in amount somewhat greater than is readily and quickly taken up, the excess serving to produce hypochlorite on subsequent addition of alkali. I now add sufficient alkali to make the mixture alkaline and convert the residual chlorine into hypochlorite. Because of the economies in alkali consumption achieved in the present process, it is quite feasible here to use caustic soda in lieu of lime. In most pulp bleaching operations where alkali is to be added after the introduction of chlorine, the amount required makes the use of caustic alkali impracticable because of cost considerations. The alkalinized mixture of the present invention is then allowed to stand for, say, half an hour to exhaust the available chlorine. The liquor is removed and the fiber washed. The liquor is yellow in color and the washed pulp is very light in color.

In this second introduction of chlorine I sometimes lessen the amount somewhat and drain and wash the fiber. This takes away acid matter and impurities. The washed fiber is then re-converted into a suspension and the rest of the chlorine and alkali added.

The process can be considered complete at this

point, but as a rule I complete the treatment with an ordinary mild bleach, usually with calcium hypochlorite.

The paper made from the bleached pulp of the present invention is very light in color, but has almost the strength and toughness of unbleached fibers. The present process is particularly applicable to the dark "hard bleaching" kraft pulps which ordinarily lose considerable strength in complete bleaching.

The feasibility of using caustic soda in the present process is of considerable practical importance. A caustic soda alkalinity is better than a lime alkalinity. When lime is used subsequent to chlorine treatments of pulps in quantity sufficient to neutralize all the acidity and give an excess, the resulting lime compounds are sometimes difficult to wash from the pulp. No such disadvantage obtains with soda. However, lime can be used in my process much more successfully than in prior processes in which chlorination takes place in a single step. Much less is required and I have discovered that most of the material responsible for the formation of insoluble compounds with lime, is removed in the first liquor. This makes it simple to use lime at the end of the second chlorination and the resulting pulp is easy to wash.

In a specific example of a process within the purview of this invention, there was taken a ton batch of a dark kraft pulp which was difficult to bleach by ordinary procedures. The pulp was converted into a 3.5 per cent aqueous suspension (70 pounds of fiber, air-dry basis, in the ton of suspension). To one ton of this pulp was introduced 4 per cent of chlorine gas (80 pounds of chlorine) during the course of 10 minutes. The mixture was then left standing 1 hour to exhaust the available chlorine. At the end of this time the liquor was separated and the pulp fiber washed thoroughly with water. The off-liquids were strongly acid. About 2 per cent of organic matters and 2 per cent of acid, were removed in this step.

In the next step the pulp fiber, again converted into a 3.5 per cent suspension, was treated with 2 per cent of chlorine (40 pounds of chlorine) and a period of 10 to 15 minutes allowed for the chlorine to act. At the end of this time sufficient caustic soda was added to make the mixture alkaline; in this example, 3 per cent (60 pounds) commercial NaOH. Lime could be substituted, in which case 3.2 per cent of lime (64 pounds of commercial hydrated lime) would be employed. The mixture was allowed to stand for 30 minutes to exhaust the available chlorine, the liquor removed and the fiber washed with water. The liquor and washings were yellow in color.

The washed pulp, though now quite light in color, was then finished by subjection to an ordinary mild bleach, with an alkaline bleach liquor, as extreme lightness was desired. The bleach (with calcium hypochlorite) liquor required only 1.5 per cent available chlorine (30 pounds per ton). The final product made into paper was very light in color, but preserved the strength and toughness of the unbleached fibers.

In this example, the total amount of chlorine used was 150 pounds per ton of 3.5 per cent suspension, as compared with the 180 pounds per ton, which would be required if all the chlorine be added in a single stage. The amount of alkali (using lime) was 108 pounds as compared with 226 pounds necessary in the conven-

tional procedures. By interpolating a washing step between the second chlorination and the addition of alkali, the consumption of alkali can be reduced still further.

5 The process of the invention has been found well adapted for treatment of various kinds of kraft pulps including grades considered easy, relatively difficult and very difficult to bleach. The stated advantages are obtained with all these
10 pulps. Also, the process is applicable to pulps other than kraft, the same advantages resulting to a greater or less degree.

What I claim is:—

15 1. A method of bleaching pulp which comprises treating an aqueous pulp suspension containing not over approximately 4 per cent pulp, with free chlorine, in amount insufficient for

complete chlorination of the non-cellulose impurities, removing the liquor, washing with water, reconvertng the fiber into a thin aqueous suspension of concentration not greater than that of the first suspension, treating with additional
5 chlorine in excess of the amount required for direct chlorination of the remaining impurities, making the pulp alkaline so as to form hypochlorite from the residual chlorine, allowing time
10 for hypochlorite bleaching to take place, removing liquor, washing and bleaching with hypochlorite liquor.

2. The process of claim 1 wherein the pulp suspension is made alkaline with caustic soda.

3. The process of claim 1 wherein the pulp
15 suspension is made alkaline with lime.

FERRI CASCIANI.