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BLEACHING OF KRAFT PULP

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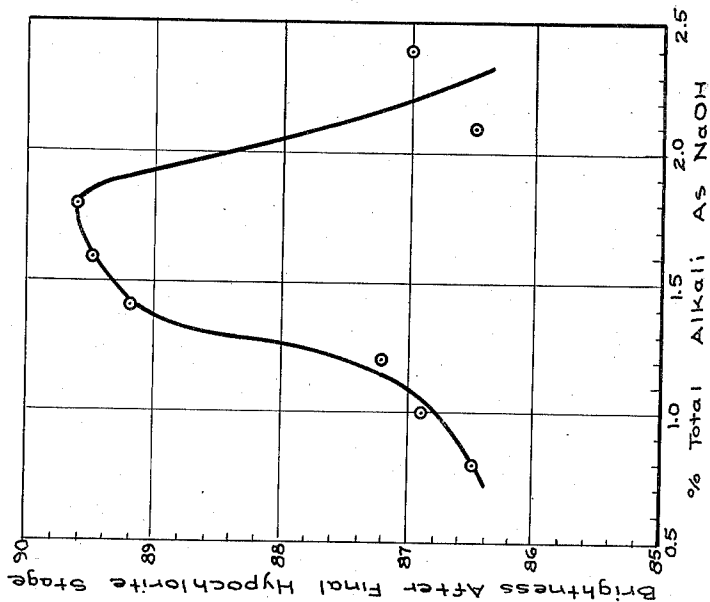


FIGURE 2
Effect Of Alkalinity In
Caustic Extraction Stage
On Brightness After
Hypochlorite Stage

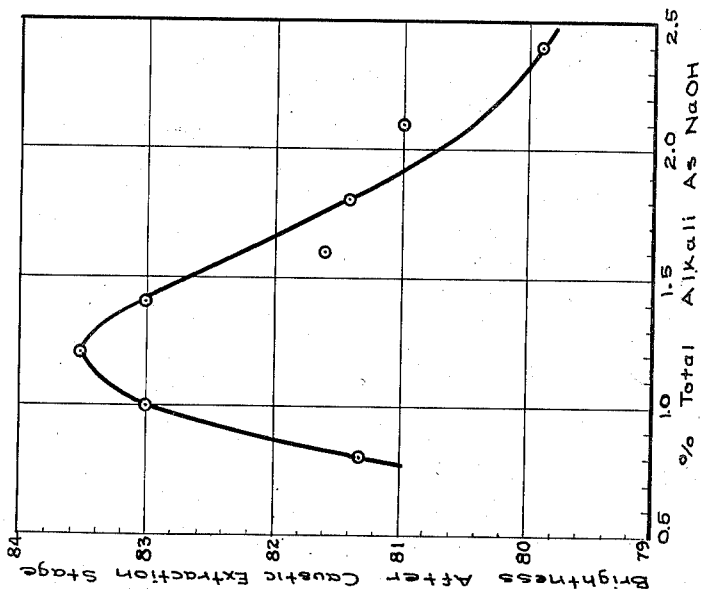


FIGURE 1
Effect Of Alkalinity In
Caustic Extraction Stage
On Brightness After
Caustic Extraction Stage

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BLEACHING OF KRAFT PULP

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4 Claims. (Cl. 8—106)

This application relates to the bleaching of wood pulps and particularly to multi-stage processes for bleaching kraft pulp.

Various multi-stage processes for bleaching kraft pulp have been proposed, most of which involve a series of treatments with one or more oxidizing agents. The more conventional processes generally start with a purification treatment using chlorine or hypochlorite, generally the former or both, in one or more stages, after which impurities which have been solubilized are washed out with water or more frequently with an alkaline solution. When using chlorine, the alkali may be added while a substantial amount of chlorine is present, in which case hypochlorite is formed in situ and is permitted to react with the pulp. After such preliminary purification, the pulp is generally partially bleached with hypochlorite, washed and then subjected to a final bleach with hypochlorite. The pulp is frequently subjected to one or more caustic extraction treatments inserted between the treatments indicated. Thus, a caustic extraction following the preliminary chlorination and preceding the first hypochlorite stage is quite common, as is also a caustic extraction between the last two hypochlorite stages.

Multi-stage processes of the above general type have also been proposed in which a peroxide bleach is employed as the final stage. It has also been proposed (Luth U. S. P. 2,030,384) to insert between successive hypochlorite stages, a caustic extraction treatment employing a caustic soda solution containing sodium peroxide. Such a process appears never to have achieved any significant commercial use, because the conditions suggested in the patent do not make the use of peroxide practical. The present invention relates generally to such a process and is based upon the discovery that when employing peroxide in such a caustic extraction treatment, the benefits derived from the treatment are highly dependent upon the amount of alkali employed.

It is an object of the invention to provide an improved method for bleaching kraft pulp. A further object is to provide an improved multi-stage process for bleaching kraft pulp which includes a number of treatments with active chlorine agents and also an intermediate caustic extraction treatment using an alkaline liquor containing a peroxide. A particular object is to provide a process in which the alkalinity of the liquor employed in such a caustic extraction treatment is adjusted to a value within a range which has been found to be highly critical. Other objects will be apparent from the following description.

It has now been found that in multi-stage processes for bleaching kraft pulp in which active chlorine agents are employed in a series of treatments and which include an intermediate caustic extraction treatment using an extracting liquor containing a peroxide, the final brightness of the treated pulp is highly dependent upon the amount of alkali present in the treating liquor employed in such extraction stage. More particularly, it has been found that maximum final brightnesses are achieved when the total alkalinity of the liquor employed in such an inter-

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mediate extraction with a liquor containing peroxide, is within the range of 1.0 to 2.2%, calculated as NaOH and based on the dry weight of the pulp. However, at least 60% of the total alkalinity should be derived from an alkali metal hydroxide or peroxide added as such. Accordingly, the objects of the invention are accomplished by employing a caustic extraction liquor in an intermediate treatment of a multi-stage process of the above type, which liquor will have a total alkali content within the range stated with at least 60% of said total alkali being derived from an alkali metal hydroxide or peroxide, or both.

The above optimum alkalinity range for the caustic extraction stage, which stage is to be followed by a treatment with an active chlorine agent such as a hypochlorite, has been found to be different from the optimum alkalinity range for a final peroxide bleaching stage. In other words, if a caustic extraction treatment with a liquor containing peroxide is to be employed as the final treating stage, the alkalinity which is optimum for that treatment has been found to be different from that which is optimum for the extraction treatment, if the extraction treatment is to be followed by a hypochlorite treatment. This discovery is surprising to say the least, since it normally is expected that the brightest pulp from the extraction stage would give the brightest pulp after a subsequent hypochlorite bleach. Such, however, is not the case.

In the experiments described below, and elsewhere in the specification, all percentages of reagents stated are percentages by weight and all are based upon the dry weight of pulp being treated, unless stated to be otherwise. The pulp consistencies reported represent the percentages by weight of pulp, calculated as dry pulp, present in the mixtures of pulp and treating liquors indicated. All brightness values reported were determined using a Hunter reflectometer on air-dried pulp handsheets prepared as described in Tappi procedure No. T.205m-50.

A southern pine kraft pulp having an initial brightness of about 21 was subjected to a preliminary chlorination at 4% consistency and 73° F. for 30 minutes using 4.35% chlorine, equivalent to 55% of the chlorine demand of the pulp. After thorough washing, the pulp was treated for 1 hour at 175° F. and a consistency of 15% with 3% caustic soda. The pulp was washed then partially bleached by treatment during 2 hours at 100° F. and a consistency of 15% with 1.04% chlorine as sodium hypochlorite, which amount corresponded to 90% of the chlorine demand. After a thorough wash, the pulp had a brightness of 72.6.

Individual samples of the pulp purified by the above treatments were subjected to caustic extraction treatments with liquors containing 0.42% H₂O₂, 3.0% of 41.6° Bé. sodium silicate (8.9% Na₂O, 29% SiO₂ and 62.1% H₂O by weight), 0.05% Epsom salt and sufficient added sodium hydroxide to give total alkalinities, calculated as NaOH and based on the dry weight of the pulp, equal to the values shown in the table below. In each instance the caustic extraction treatment was carried out during 2 hours at a consistency of 15% and at 180° F. The brightnesses of the caustic extracted samples are shown in the table.

A portion of each pulp sample which had been caustic extracted with alkaline liquor containing peroxide as indicated above was washed thoroughly, subjected to a final hypochlorite bleach for 1.5 hours at 15% consistency and 100° F. using sodium hypochlorite equivalent to 0.5% chlorine, and then thoroughly washed.

The table shows: in column (1), the sample number; in column (2), the total alkali content (as NaOH) of the caustic extraction liquor containing peroxide used in the treatment just preceding the final hypochlorite bleach; in

column (3), the brightness immediately following the caustic extraction treatment; and, in column (4), the brightness following the final hypochlorite bleach.

Table.—Effect of total alkalinity in caustic extraction stage on brightness

Sample (1)	Percent Total Alkali in Caustic Extraction Stage (2)	Brightness After—	
		Caustic Extraction Stage (3)	Final Hy- pochlorite Stage (4)
1.....	0.8	81.3	86.4
2.....	1.0	83.0	86.9
3.....	1.2	83.5	87.2
4.....	1.4	83.0	89.2
5.....	1.6	81.6	89.5
6.....	1.8	81.4	89.6
7.....	2.1	81.0	86.5
8.....	2.4	79.9	87.0

¹ Maximum brightness.

The curve of Fig. I is a plot of the brightnesses in column (3) against the alkalinities in column (2), while the curve of Fig. II is a plot of the brightnesses in column (4) against those same alkalinities.

It is evident from the above data and the corresponding curves that the total alkalinity of the extracting liquor containing peroxide is critical whether or not the extraction treatment constitutes the final treatment. It is also clearly evident that the critical range for total alkali in the extraction treatment, when that treatment is to constitute the final treatment, is quite different from the critical range when the extraction treatment is to be followed by a hypochlorite bleach. In the former case, the critical range is 0.8 to 1.8%, the optimum alkalinity being 1.2%; while in the latter case, the critical range is 1.0 to 2.2% and the optimum alkalinity is 1.8%. This difference was entirely unexpected.

In a similar series of experiments it was found that neither the critical alkalinity range nor the optimum alkalinities for use in the extraction treatment with liquor containing peroxide, varies to any substantial extent as the time of the extraction treatment is varied from 1 to 3 hours. Also, it was found that the presence or absence of silicate in the extracting liquor is not critical, particularly as regards the brightness after the subsequent hypochlorite bleach, so long as the amount used is not equivalent to more than about 40% of the total alkalinity present. However, the presence of sodium silicate in the extracting liquor in amounts corresponding to 0.5 to 5% of the pulp weight is generally preferred in view of its known stabilizing action, particularly where the alkaline peroxide solution is to be stored for any appreciable time before use. Stabilizers other than silicate, e. g., sodium pyrophosphate, can also be used.

It was also demonstrated in a similar series of experiments that at alkalinities higher (up to 5%) and lower (down to 0.4%) than those within the critical ranges, substantially lower brightnesses result both after the extraction stage and after a subsequent hypochlorite stage.

The conditions, such as pulp consistency, temperature, amount of chlorine or hypochlorite, etc., for carrying out the preliminary purification treatments and the partial bleaches with hypochlorite may be varied considerably and any condition commonly known to be suitable can be used. The order and number of the treatments can be varied and usually will be governed by the extent of purification and bleaching desired. The procedure illustrated in the experiments described above involving a preliminary chlorination at low consistency (e. g. 2 to 5%), a caustic extraction stage at medium consistency (10 to 20%) using about 2 to 4% caustic soda, and a partial bleach at medium consistency with an alkali metal or an alkaline earth metal hypochlorite, gives excellent

preliminary purification of the pulp. However, multiple chlorinations or partial bleaches with hypochlorite can be used. Good results can also be obtained using a modified chlorination procedure in which the pulp suspension is rendered alkaline before all of the added chlorine has been consumed so as to form hypochlorite in situ.

The treatment with an active chlorine agent, such as sodium or calcium hypochlorite, which follows the alkaline extraction with liquor containing peroxide, can be carried out in one or more stages and can be followed, if desired, by other treatments or combinations of treatments, e. g., by a peroxide bleach. However, a single hypochlorite bleach, preferably at medium consistency and at a temperature of 80 to 120° F., employing an amount of hypochlorite equal to 0.3 to 1.5% of chlorine, based on the pulp weight, will generally constitute the final treatment.

The alkaline extraction treatment with liquor containing peroxide, which is to be applied intermediate two treatments with active chlorine agents and which usually will precede a final hypochlorite bleach, will preferably be carried out employing liquor containing a peroxygen compound in an amount equivalent to 0.1 to 1.0% H₂O₂, based on the pulp weight. Hydrogen peroxide or any recognized equivalent peroxygen compound, e. g., the alkali metal peroxides, perborates, perphosphates, percarbonates, etc., can be used. Greater or smaller amounts of the peroxygen compound, e. g., as low as 0.03% and up to 2.0% H₂O₂ can be used. Amounts smaller than about 0.03% are generally too low to be beneficial while amounts greater than about 2.0% usually result in no added advantages.

The above extraction treatment is preferably carried out at a pulp consistency of 10 to 20% although higher or lower consistencies can be used. Somewhat longer times are usually desirable at lower than at higher consistencies. The desired effect of the treatment is obtained more rapidly at higher than at lower temperatures. The preferred temperatures are in the range 160 to 200° F. Lower temperatures, e. g., as low as room temperature, are effective if a longer time is not objectionable. Temperatures substantially above 200° F. can be used but are not recommended because of the relative instability of alkaline peroxygen solutions at such temperatures. Under the preferred conditions of consistency and temperature, a treating time of 0.5 to 2 hours is usually satisfactory.

All alkalinities reported herein are expressed as percentages by weight NaOH based on the dry weight of the pulp and have reference to the initial alkalinity of the treating liquor. In expressing the alkalinities in terms of NaOH, it is not to be understood that sodium hydroxide is the only alkaline material to be used. On the contrary, any suitable commonly used alkalinizing agent, or combination of such agents, can be employed as indicated below, it being understood that the total alkalinity value will take into account all alkaline materials present. The total alkalinity can be derived entirely from the addition of an alkali metal hydroxide or peroxide, or both; and at least 60%, preferably 80 to 90%, will be derived from that source. Up to 40% of the total alkalinity can be derived from other commonly used alkaline materials such as the alkali metal carbonates, phosphates, silicates, borates, etc., or the alkaline earth metal hydroxides, or mixtures thereof. The total alkalinity in terms of sodium hydroxide can be readily determined by standard acid titration methods employing, for example, phenol red as the end point indicator.

The presence of a peroxygen compound in the liquor for the caustic extraction has a two-fold effect; it bleaches and at the same time increases the effectiveness of the caustic extraction. These desirable effects are not realizable to the desired maximum extent unless the total alkalinity of the liquor is adjusted to within the critical range defined above. When the total alkalinity is within that range, the use of peroxide makes possible the obtaining of high quality pulp, e. g., in the brightness range

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of 85 to 90. The peroxide causes no significant degradation of the pulp or loss in pulp yield and reduces the amount of hypochlorite required in the subsequent bleach to give high brightness pulp.

I claim:

1. In a multi-stage process for bleaching kraft pulp in which active chlorine agents are employed in a series of treatments including a final hypochlorite bleaching stage and which includes an intermediate caustic extraction treatment using an alkaline extracting liquor containing a peroxygen compound prior to said final hypochlorite bleaching stage, the improvement comprising employing in said extraction treatment a liquor having a total alkali content of 1.0 to 2.2% calculated as NaOH and based upon the dry weight of the pulp, at least 60% of said total alkalinity being derived from a material of the group consisting of the alkali metal hydroxides and alkali metal peroxides.

2. The process according to claim 1 wherein the extracting liquor contains a peroxygen compound in an amount corresponding to 0.03 to 2.0% H_2O_2 based upon the dry weight of the pulp.

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3. The process of claim 1 wherein the total alkali content is about 1.8%.

4. The process of claim 2 wherein the total alkali content is about 1.8%.

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