

## UNITED STATES PATENT OFFICE

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## TREATMENT OF GROUND WOOD PULP

Robert L. McEwen, Williamsville, N. Y., assignor  
to Buffalo Electro-Chemical Company, Inc.,  
Tonawanda, N. Y.

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4 Claims. (Cl. 8—106)

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The present invention relates to an improvement in the treatment of mechanical pulp such as that derived from the grinding of wood. The invention relates particularly to treatment of such mechanical or ground wood to improve its brightness and therefore increase the field of use of such product in the manufacture of paper. The present application is a continuation of Serial No. 572,255 filed January 10, 1945, now abandoned.

Wood which is ground under pressure at an elevated temperature by abrading contact with a grinding stone has been generally designated "mechanical ground wood pulp" or "ground wood pulp," whether the pulp be obtained from the log as cut and debarked or from logs first subjected to preliminary steaming, water soaking in acid, alkaline or salt solution prior to the grinding operation. Until within recent years, mechanical ground wood pulp was employed only in the manufacture of lower grade paper items due to the fact that the pulp did not lend itself readily to brightening operations. Ground wood pulp is superior to chemical pulp for all types of printing papers and papers requiring high opacity and good bulk.

Unlike chemical pulps, such as the well-known soda, kraft or sulfite pulps, ground wood pulp is not amenable to the usual chlorine or hypochlorite treatments so universally employed for brightening chemical pulps. On the contrary, it is well known in the pulp and paper industry that chlorine treatment of ground wood pulp darkens the pulp rather than lightens it, probably by reason of the formation of chlorolignins or other colored chlorine substances.

Some success has been obtained in brightening ground wood pulp by treating such pulp with alkaline peroxide solutions. Although the degree of brightness is not exceptional the resultant paper finds a better market than paper made from untreated ground wood pulp. However, the treatment is costly since the consumption of active oxygen is high for the slight degree in brightness obtained.

It is an object of the present invention to provide a commercially suitable method for brightening mechanical wood pulp to overcome the disadvantages inherent in the prior process. It is a further object to provide a more efficient and more economic method for brightening mechanical wood pulp. It is also an object of the present invention to provide a method of brightening mechanical wood pulp with active oxygen, that is to say, peroxide solutions, whereby a greater de-

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gree of brightness may be obtained from a given amount of peroxide. It is a further object of the invention to provide a preliminary treatment of mechanical wood pulp whereby a more uniform treatment of such pulp is effected by active oxygen.

In accordance with the present invention it has been found that exceptional brightening of mechanical wood pulp may be obtained with peroxide solutions provided the ground wood pulp is first given a preliminary treatment with active chlorine, such as a hypochlorite, even though such treatment results in an initial darkening of the pulp. The subsequent peroxide bleach produces a greater degree of brightness than the same peroxide treatment upon mechanical wood pulp not so treated.

In general, the pulp, formed in any suitable manner, whether directly from logs as cut, from debarked or from soaked logs, is screened and concentrated to a desired consistency which is generally in the neighborhood of 2% to 6%, more or less, that is, usual mill consistencies, although the process is operable at higher consistencies. The pulp is then treated at room or slightly elevated temperatures with an active chlorine solution under a controlled degree of pH and active chlorine concentrations. As has been the universal experience with the treatment of mechanical pulps by chlorine reagents, the pulps are not lightened in color but are darkened substantially. Thereafter the pulp is brightened to the desired degree by the employment of an alkaline peroxide solution, the treatment being effected at room or moderately elevated temperatures.

The active chlorine employed may be slightly acid, neutral or slightly alkaline solutions of hypochlorite. The solutions vary in pH from about pH 3.5 to 9.0, more or less. In general the preferred range will be between about pH 5 to 7.5. Generally alkali metal hypochlorites are the preferred reagent although alkaline earth metal hypochlorites may serve the purpose. In some cases, chlorine gas is bubbled through the slurry, provided suitable agitation and pH control are obtained. The active chlorine concentration in the pulp suspension calculated on the basis of dry pulp may vary from 0.1% to 1.75%; generally a concentration of 0.25 to 1.0% active chlorine will suffice.

The alkaline peroxide solution may be prepared from hydrogen peroxide and substantially any alkali or from the alkaline peroxides such as alkali metal peroxide, alkaline earth peroxides as well as perborates and the like. The alkaline

peroxide solution is stabilized in any well-known manner as, for instance, by the employment of silicate of soda. The peroxide concentrations may vary from 0.25% to 10%  $H_2O_2$  100 volume, dry basis, although 1% to 2%  $H_2O_2$  100 volume will be most generally employed.

The following examples will serve to illustrate the preferred mode of operation; it will be understood that there are many variations possible, for instance, consistencies of pulp, pH of pulp suspension, concentration of reagents employed, temperature, and time of treatment depending upon the pulp stock employed and the end result desired.

To show the effect of alkaline peroxide treatment directly upon mechanical pulp, a sample of ground wood pulp of 2.55% consistency and having an initial brightness of 66.0 units, as determined by the Higgins photoelectric colorimeter, was subjected at 40° C for 6 hours to the action of an alkaline peroxide solution containing 2% 100-volume hydrogen peroxide, 8% silicate of soda, specific gravity 1.42, and 1.3% sodium hydroxide, all based on oven dried weight of pulp. The pulp slurry was then acidified to a pH of 5 and paper sheets prepared and the color determined as 69.8 units, an increase of 3.8 units.

Another portion of the same mechanical pulp was subjected first to a preliminary treatment by contact for 10 minutes with a solution containing 0.5% active chlorine, dry basis of pulp, at a pH of 6.0 at room temperature. The pulp so treated showed a lessening of brightness as a sample tested for brightness possessed a color of but 65.0 units, i. e., a decrease of 1 unit. The chlorine treated pulp was thereafter subjected to a peroxide treatment identical with that described immediately above. After the peroxide treatment the pulp was acidified to a pH of 5 and paper prepared from the pulp. The resultant paper possessed a brightness of 74.3 units. This is in contrast with peroxide alone which produced a color of 69.8 units.

Mechanical pulp of an initial brightness of 66.0 units was subjected to an alkaline peroxide treatment, as described in Example #1, and a brightness of 70.3 units was obtained. Another portion of the same pulp was first subjected for 15 minutes to the action of 0.5 active chlorine, in weight of the pulp, dry basis, at a pH of 5.9 and then immediately to an alkaline peroxide solution containing 2% hydrogen peroxide 100-volume, 8% sodium silicate and 1.3% sodium hydroxide for six hours, based on oven dried pulp. After acidification the resultant paper sheet showed a brightness of 74.6 units. A sample of the chlorine treated pulp possessed a brightness of only 64.8.

The pretreatment of ground wood pulp with active chlorine permits the fullest employment of the active oxygen content of the alkaline peroxide solution. Pulp so preliminarily treated may be brightened to a greater degree with a given amount of peroxide than pulp not so preliminarily treated, probably by reason of the fact that the peroxide bath is more stable in the presence of active chlorine treated pulp. This phenomenon is illustrated in Example 3 as follows:

Ground wood pulp, having an initial brightness of 66.0 was subjected at a consistency of 3.14% to the action of a peroxide bleach bath containing 2.0% hydrogen peroxide 100-volume, 8% silicate of soda, 1.3% sodium hydroxide all on an oven-dry pulp basis for a period of six

hours at 40° C. After acidification of the pulp suspension to a pH of 5, the resulting brightness was 69.6 brightness units. After three hours of this six-hour bleaching treatment, all of the hydrogen peroxide had been consumed.

Another portion of the same pulp was first treated for 15 minutes with 0.5% active chlorine on an oven dried pulp basis at a pH of 5.9 and then subjected to the same peroxide bleach as noted immediately above. After acidification the brightness of the paper formed from the pulp suspension was 74.6 units. The active oxygen present at the end of the six-hour bleaching treatment was 31% of that originally employed. This comparison of oxygen stability will be more readily seen from the following table, the pH and total alkalinity of each bath being substantially identical throughout the period of test:

	Pulp Subjected to Peroxide Bleach Alone, Per Cent Hydrogen Peroxide Left	Pulp Subjected to Peroxide Bleach Subsequent to Chlorine, Per Cent Hydrogen Peroxide Left
Original.....	100	100
After 1 hour.....	43	55
After 3 hours.....	0.0	39
After 6 hours.....	0.0	31

The amount of chlorine employed, calculated on the basis of the dry pulp, should not be too great, otherwise the darkening of the pulp proceeds to such an extent as to minimize the subsequent action of the peroxide. Thus ground wood pulp of a brightness of 66.0 was treated for 15 minutes at pH 5.8 and a consistency of 2.72 with 2% active chlorine on the dry pulp basis. Thereafter the pulp was brightened with the 2% hydrogen peroxide 100-volume treatment already described. Paper prepared at pH 5 from this pulp possessed a brightness of 66.3.

Where the pH of the pulp suspension during the active chlorine treatment is too low, little or no improvement in brightness is observed. In other words, there appears to be little or no cooperative action between the active chlorine treatment and the alkaline peroxide treatment as described heretofore. A mechanical pulp at 3.0% consistency treated for 30 minutes at pH 1.8 using 0.5% active chlorine on a dry basis, and thereafter treated with 2% alkaline peroxide as described, increased the brightness from 66.0 to 68.5 as contrasted with peroxide alone which gave a brightness of 71.0. When the chlorine treatment was performed on the same pulp at pH 9.2 the brightness after peroxide treatment was 71.9.

In accordance with the present invention therefore it has been found that it is beneficial to maintain the pH of the hypochlorite solution in which the pulp is suspended between 3.5 and 9 although the preferred range is between 5 and 7.5. The time of treatment depends to a certain extent on the type of the pulp, on the per cent of active chlorine used for treatment, and on the pH, temperatures, and on the consistency employed. In general, however, a treating time of 10-45 minutes is preferred at room or slightly elevated temperature with active chlorine of 0.25% to 1% dry basis preferred.

While the consistencies given in the examples above range from 2.55 to 3.05%, other higher or lower consistencies may be employed. In fact, higher consistencies are preferred since even greater brightness may be obtained with the same

percentage of chemicals based on dry pulp. The best effect is obtained by subjecting the mechanical pulp first to the above described hypochlorite treatment for a period of time insufficient to allow complete consumption of the active chlorine and long enough to obtain the desired conditioning effect. As described in Example #2, an even greater brightness is obtained under these preferred operating conditions.

The term "active chlorine" as used herein, designates an aqueous solution containing a hypochlorite or chlorine; such a solution may be prepared by passing chlorine gas into water and the pH raised with alkali or by passing chlorine into aqueous alkali and the pH regulated with acid; or the solution may be prepared from water soluble hypochlorites including hypochlorous acid.

The alkaline peroxide solution may be prepared with hydrogen peroxide and alkali or with any other peroxide such as sodium peroxide, sodium perborate, etc. Other stabilizers or alkalies may be employed in lieu of or to replace sodium silicate or the sodium hydroxide described above.

The surprising result described herein is due, it is believed, to inactivation of certain catalysts detrimental to alkaline peroxide solutions which occur naturally in the pulp. It should be especially noted that the brightness of the pulp, after the active chlorine treatment, is lower than the original but that the subsequent peroxide treatment not only overcomes this decrease in brightness but, in addition, produces a brightness after peroxide treatment greater than that obtained without the chlorine pretreatment.

What is claimed is:

1. The method of producing bleached mechanical wood pulp which comprises first treating the pulp at a pH in the range pH 3.5 to 9 with a solution of a hypochlorite sufficient to produce in the pulp an active chlorine content of 0.1% to 1.75% based on the weight of dry pulp and thereafter with an alkaline peroxide solution to brighten the pulp to the desired degree.

2. The method of producing bleached mechanical wood pulp which comprises first treating the

pulp at a pH in the range pH 3.5 to 9 with a solution of a hypochlorite sufficient to produce in the pulp an active chlorine content of 0.1% to 1.75% based on the weight of dry pulp and thereafter with 0.25% to 10% alkaline peroxide calculated as 100 volume  $H_2O_2$  on the weight of the dry pulp.

3. The method of producing bleached mechanical wood pulp which comprises first treating the pulp at a pH of 5 to 7.5 in an aqueous solution of a hypochlorite containing 0.25% to 1.75% active chlorine based on the weight of dry pulp and thereafter with sufficient alkaline peroxide to brighten the pulp.

4. The method of producing bleached mechanical wood pulp which comprises first treating the pulp at a pH in the range pH 3.5 to 9 with a solution of a hypochlorite sufficient to produce in the pulp an active chlorine content of 0.1% to 1.75% based on the weight of dry pulp and thereafter with an alkaline peroxide solution containing less than 10% peroxide calculated as 100 volume hydrogen peroxide based on the dry weight of the pulp.

ROBERT L. McEWEN.

#### REFERENCES CITED

The following references are of record in the file of this patent:

#### UNITED STATES PATENTS

Number	Name	Date
848,361	Gagedois	Mar. 26, 1907
1,768,820	Bradley	July 1, 1930
1,953,191	Richter	Apr. 3, 1934
2,147,618	Rawling	Feb. 14, 1939
2,187,016	Craig	Jan. 16, 1940
2,359,891	Brallier	Oct. 10, 1944

#### OTHER REFERENCES

40 Reichert et al., Mills et al., "Peroxide Bleaching of Mechanical Pulps—Influence of Bacteria and Enzymes," Paper Trade Journal, Jan. 31, 1946, Tappi Section pages 41-44; May 16, 1946, Tappi Section pages 216-220.