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BLEACHING OF KRAFT PULP BY MEANS OF HYDROGEN PEROXIDE. THE EFFECT OF ACID TREATMENT AND CALCIUM CHLORIDE TREATMENT BEFORE THE LAST STAGE.

Reported by

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SUMMARY

From the results of the experimental work conducted, it appears that hydrochloric acid pretreatment of chlorinated and caustic extracted, and, also, chlorinated, caustic extracted, and calcium hypochlorite bleached kraft pulp before last stage hydrogen peroxide bleaching was not beneficial to final brightness.

Calcium chloride pretreatment, on the other hand, was definitely of value to final brightness in three stage bleaching. However, calcium chloride pretreatment of chlorinated, caustic extracted, and calcium hypochlorite bleached kraft pulp did not increase pulp brightness to a significant extent.

It seems, therefore, that there was an optimum concentration of calcium salts which was of maximum benefit to brightness.

Also, there was some indication that both hydrochloric acid and calcium chloride pretreatment of the calcium hypochlorite treated pulp caused small increases of reflectance in the yellow-orange range of the spectrum.

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INTRODUCTION

Historical Review

One of the earliest agents used for bleaching of wood pulp was bleaching powder and bleach liquor containing calcium hypochlorite. This chemical was discovered by Charles Tennant of Scotland in 1798. His development of the reaction of chlorine gas with milk of lime was outstanding from the economical point of view. The use of calcium hypochlorite as a bleaching agent made possible much higher brightness than had been previously possible. Calcium hypochlorite has continued to be an important bleaching agent to this date.

Burgess and Watt (1), in 1854, developed a method of direct chlorination for bleaching of soda pulp, but their effort was not a commercial success due to the high cost of elemental chlorine and the absence of corrosion resistant equipment. (2)

The next development came in 1895, when the Bellmer brothers in Germany developed new equipment suitable for bleaching at an increased consistency of about seven percent. Increased brightness and improved chemical efficiency was observed as a result of the relatively high density. This trend towards high densities continued, and soon bleaching at densities of about 15 to 25 percent was possible due to the development of new equipment. (3)

Further improvement was made by Cross and Bevan. They demonstrated that when chlorine reacted with lignin, compounds were formed which were soluble in caustic soda. This led to the use of chlorine not only as an oxidizing agent but, also, as an agent which rendered the lignin soluble, making possible its caustic extraction. It was also noted that diminished damage was done to the cellulose by this method. (3)

It was not until a few years before World War one that a commercial bleaching method of direct chlorination was developed. At this time, de Vain (4) obtained patents for a method of bleaching sulfate pulp in which chlorination was the first stage in a multi-stage process. Drewsen (5), in 1918, evolved a similar process. These processes were developed commercially after World War one due to the reduced price of liquified chlorine and the development of railroad tank cars suitable for its transportation.

At this same time, plant scale multi-stage processes were developed. It was discovered that multi-stage processes reduced chemical consumption or gave increased brightness. The number of stages increased to as many as eight or nine before long and gave the increased brightness desired. To prevent degradation of the cellulose in the last stages, new bleaching agents were developed, such as chlorine dioxide and the alkali peroxides, that would not harm the fiber but, would give the desired brightness. The tendency changed from eight or nine stages to four or five stage bleaching due to the ability of these new bleaching agents to bleach to high brightness without significant damage to the fiber. Savings in capital investment were made in some cases by simplified design of new plants.

The difference in the degree of degradation of ~~to~~ the fiber may be explained by means of the oxidation potentials characteristic of these various bleaching agents. It seems that all bleaching agents whose oxidation potential is above 0.9 volts attack the cellulose fiber and that the bleaching agents whose oxidation potential is below 0.9 volts do not cause any damage to the fiber. (2) This may be seen from figure 1. Valko (6) states, however, that the oxidation potential of hydrogen peroxide in reference to the normal hydrogen potential is minus 1.77 volts. This seems to indicate that hydrogen peroxide is one of the strongest oxidizing agents, and that its gentle action is probably due to rate factors rather than equilibrium condition.

In 1936, Luth, Nugent, and Christian (7) developed a process for bleaching sulfate pulp by means of an alkali metal peroxide treatment. This was one of the first developments in the use of peroxides in the bleaching of wood pulp. The use of peroxides was investigated further, and in 1944, the use of epsom salt and pyrophosphates as stabelizing agents was recommended. (8, 9)

Recent Developments

In recent years, peroxide bleaching has been extensively developed, especially for groundwood and semichemical pulps. Peroxide bleaching of these pulps has been successful and has resulted in increased brightness, good color stability, and hardly any degradation to the cellulose.

The most recent development in peroxide bleaching has been the pretreatment of the pulp before the last stage peroxide bleach. In the multi-stage bleaching of kraft pulp, using peroxide for the last stage, it was found to be advantageous to treat the pulp with an acid preceding the last stage if lime and/or calcium hypochlorite were used in preceding stages. Hydrochloric acid or chlorine may be used with equivalent results. The purpose of acid pretreatment was to remove residual lime and lime impurities which were said to cause wasteful decomposition of the alkaline peroxide solutions. (9, 10)

The result of acid pretreatment has been improved peroxide bleaching efficiency in some instances by as much as five to ten times. A commercial bulletin states that the improvement obtained by using the acid pretreatment in the peroxide bleaching of lime treated kraft pulp is greater than that previously obtained by varying any of the normal bleaching conditions, i. e., time, pH, peroxide concentration, temperature, or pulp consistency. It was claimed that this process produced bleached kraft with a G. E. brightness of 88 in addition to good strength retention and good color stability. (10)

It has also been established that improved final bleached pulp brightness may be obtained by pretreating groundwood pulp prior to peroxide bleaching. (11, 12) In this case, calcium chloride, added at the rate of 20 to 30 pounds per oven dry ton of pulp, has reportedly resulted in brightness gains of 0.5 to 2.0 points. (13, 14)

Acid pretreatment prior to peroxide bleaching of chemigroundwood has been beneficial, resulting in bleached brightness advantages of one to five points. (15)

The purpose of this research project is to make a comparison of the effect of acid treatment and calcium chloride treatment before the last stage hydrogen peroxide bleach for kraft pulp. It seems to be contradictory to remove calcium salts with acid before bleaching of kraft pulp with peroxides, and to add calcium salts before bleaching of groundwood pulp with peroxides. Therefore, it has been planned to experiment with kraft pulp after chlorination and caustic extraction, and to bleach in a third last stage with peroxides, using the two controversial pretreatment methods.

PRE-EXPERIMENTAL DATA

Hydrogen Peroxide Chemistry

Hydrogen peroxide, it has been found, does not decompose into water and atomic oxygen, but, rather, it is now generally accepted that it dissociates into ions as follows: $H_2O_2 = H^+ + (HO_2)^-$ The $(HO_2)^-$ ion is considered to be the active bleaching or oxidizing agent. From the law of mass action the $(HO_2)^-$ ion concentration may be promoted by neutralizing the hydrogen ions with an alkali, and, conversely, by the addition of an acid the $(HO_2)^-$ ion concentration may be reduced. Decomposition of hydrogen peroxide, therefore, will occur in the presence of an acid. Decomposition will also occur in the presence of heavy metals such as copper, iron, and manganesse which act as decomposition catalysts. The decomposition caused by traces of heavy metals can be retarded by use of stabilizing agents. (9)

Effect of Variables in the Hydrogen Peroxide Stage

Hydrogen Peroxide Concentration

The concentration of hydrogen peroxide is governed by the brightness desired. The usual concentration varies from 0.2 percent to 0.8 percent of 50 percent hydrogen peroxide.

Consistency

As the consistency of the pulp is increased up to approximately 16 percent, the brightness is increased and the reaction time is decreased. Increases in consistency beyond about 16 percent result in continued decrease of reaction time, but does not increase the brightness substantially.

Alkalinity

The degree of alkalinity required depends upon the degree of purification of the pulp, the temperature, and the time. If the alkalinity is too high the hydrogen peroxide will dissociate too fast, and will not give the best possible results. If the alkalinity is too low the pulp will not receive the maximum benefits of the bleaching agent. In general, it may be said that as the temperature is increased the alkalinity should be increased and the time decreased to obtain comparable results.

Temperature and Time

As the bleaching temperature is increased the rate of peroxide bleaching increases. Normal bleaching temperature for hydrogen peroxide bleaching varies from 160 F to 190 F. The time is dependent on the temperature, alkalinity, and consistency, and may vary from forty minutes to five hours. Usually a retention time of about two hours is prefered.

EXPERIMENTAL DESIGN

To arrive at significant results by experimentation, it was planned to carry out the bleaching operations in three stages, the first stage being direct chlorination, the second stage caustic extraction, and the third and final stage hydrogen peroxide bleaching. It was to be understood that each stage was to be followed by a thorough water wash to insure that the succeeding stages would not be affected by residual chemicals.

The bleaching conditions for the first and second stages were to be constant as to amount of chlorine for the first stage, amount of caustic soda for the second stage, and degree of washing after each stage. To attain uniformity, a relatively large quantity of pulp was to be chlorinated, caustic extracted, and subdivided into small portions for parallel experiments.

In some instances the conditions before and during the third stage were to be varied such as the quantity of caustic soda necessary to adjust the pH to an equal value, and the special treatments before the last stage. All other conditions during the last stage of bleaching were to be constant such as the quantities of hydrogen peroxide, of sodium silicate, and of magnesium sulfate, the temperature, time, pH, washing procedure, and method of forming brightness pads.

Results of acid pretreatment and calcium chloride pretreatment were to be compared with results of control experiments without pretreatment in order to determine the effect of the modifications on brightness and color stability.

EXPERIMENTS PERFORMED

The initial bleaching experiments consisted of first stage chlorination, second stage caustic extraction, and third stage treatment with hydrogen peroxide using quantities and modifications as indicated.

Exp. no.		Procedure
I - A	(a)	Hydrochloric acid pretreatment
	(b)	7 percent hydrogen peroxide bleach
I - B	(a)	Calcium chloride pretreatment
	(b)	7 percent hydrogen peroxide bleach
I - C	(a)	No pretreatment
	(b)	7 percent hydrogen peroxide bleach
II - A	(a)	Hydrochloric acid pretreatment
	(b)	3½ percent hydrogen peroxide bleach
II - B	(a)	Calcium chloride pretreatment
	(b)	3½ percent hydrogen peroxide bleach
II - C	(a)	No pretreatment
	(b)	3½ percent hydrogen peroxide bleach

When it became evident that three stage bleaching would not yield sufficiently conclusive results, an additional hypochlorite stage was added as third stage followed by bleaching with hydrogen peroxide as fourth stage as follows:

Exp. no.		Procedure
III - A	(a)	2 percent calcium hypochlorite bleach
	(b)	Hydrochloric acid pretreatment
	(c)	2 percent hydrogen peroxide bleach
III - B	(a)	2 percent calcium hypochlorite bleach
	(b)	Calcium chloride pretreatment
	(c)	2 percent hydrogen peroxide bleach
III - C	(a)	2 percent calcium hypochlorite bleach
	(b)	No pretreatment
	(c)	2 percent hydrogen peroxide bleach
IV - A	(a)	2 percent calcium hypochlorite bleach
	(b)	Hydrochloric acid pretreatment
	(c)	1 percent hydrogen peroxide bleach
IV - B	(a)	2 percent calcium hypochlorite bleach
	(b)	Calcium chloride pretreatment
	(c)	1 percent hydrogen peroxide bleach
IV - C	(a)	2 percent calcium hypochlorite bleach
	(b)	No pretreatment
	(c)	1 percent hydrogen peroxide bleach

EXPERIMENTAL PROCEDURE

Chlorination

In preparation for chlorination a sufficient quantity of unbleached kraft pulp was disintegrated, and screened with a laboratory flat screen. This eliminated large shives and undisintegrated pulp.

Before the pulp was chlorinated, the chlorine demand was determined by means of its permanganate number. This was done in accordance with TAPPI Standard T-214m. The chlorine demand was found to be 12.8 percent. Sixty percent of the chlorine demand was used to chlorinate the unbleached pulp.

Chlorination was carried out in an earthen crock at 3.5 percent consistency and 70 F while agitating the pulp with a variable speed mixer. After chlorination the pulp was washed and preserved with Dowicide "G".

Caustic Extraction

The chlorinated pulp was extracted with two percent sodium hydroxide based on the weight of the chlorinated pulp. The alkali was added in form of a 50 percent water solution. The consistency of the pulp during this operation was ten percent; the temperature was 160 F which was kept constant by means of a water bath. Extraction was continued for two hours, after which the pulp was washed thoroughly with tap water at 140 F. The pulp was then air dried and used for the remaining experiments.

In order to facilitate the experimental work the moisture content of the pulp was determined and 20 gm. oven dry samples were prepared.

Preliminary Peroxide Bleaching

To determine the concentration of hydrogen peroxide necessary to obtain a satisfactory brightness a group of experiments were conducted over a range of hydrogen peroxide concentrations. The formula and conditions used were the same as those outlined in Peroxide Bleaching. After bleaching the pulp was washed with distilled water and optical hand sheets were formed.

Pretreatments

Hydrochloric Acid Pretreatment

The hydrochloric acid pretreatment consisted of the following steps in the order indicated:

1. Dilution of the pulp with distilled water to three percent consistency.
2. Addition of dilute hydrochloric acid to the pulp to yield a pH value of 2.5 and retention for five minutes.
3. Thorough washing with distilled water.

Calcium Chloride Pretreatment

The calcium chloride pretreatment consisted of the following steps in the order indicated:

1. Dilution of the pulp with distilled water to three percent consistency.
2. Addition of 1.5 percent calcium chloride (anhydrous) based on the weight of the pulp in form of a water solution followed by retention for five minutes.
3. Filtering to ten percent pulp consistency.

Peroxide Bleaching

The following are the formula and conditions for all hydrogen peroxide bleaching.

Bleaching formula:	% based on o. d. weight of pulp
Epsom salt ($MgSO_4 \cdot 7H_2O$)	0.5
Sodium silicate (58.5° Be)	1.5
Sodium hydroxide (NaOH)	1.5
Hydrogen Peroxide (H_2O_2 - 50%)	variable

The ingredients of the bleaching formula were dissolved in distilled water in the order indicated and added to the pulp in solution form.

Bleaching conditions:

Consistency - - - - - - - - - - - - 10 %

Temperature - - - - - - - - - - - - 170 F

pH - - - - - - - - - - - - - - - - 11.0

All bleaching was continued until a residual peroxide concentration of two percent of the original peroxide concentration was obtained, except for the preliminary bleaching which was carried out on a time basis.

Hypochlorite Bleaching

All calcium hypochlorite bleaching was done using two percent available chlorine based on the oven dry weight of the caustic extracted pulp. The pH was kept between 9.5 and 10.0 by addition of five percent sodium hydroxide solution as needed.

Bleaching conditions:

Consistency - - - - - - - - - - - 10 %

Temperature - - - - - - - - - - - 90 F

Time - - - - - - - - - - - - - - until a negative starch iodide paper test was obtained.

Determination of Residual Peroxide

The following test was used for determination of the residual peroxide in the bleach liquor.

A sample of the pulp slurry was placed in a clean piece of cloth and 25 ml. of liquor were squeezed out into a graduated cylinder. Thereupon, 20 ml. of 20 percent sulfuric acid, 5 ml. of 10 percent potassium iodide solution, and three drops of freshly prepared saturated ammonium molybdate solution were added. Finally, 10 ml. of starch indicator were added. The mixture was titrated with N/10 sodium thiosulfate until the blue color disappeared. (9)

(1 ml. of N/10 thio = 0.00336 gm. 50 % H_2O_2)

Evaluation of Results

All optical hand sheets were formed after reducing the pH of the pulp slurry to 4.5 with dilute sulfuric acid. This made sure that results would be of comparable nature, since variations in pH would cause changes in brightness.

A color evaluation of the bleached pulp from experiment III - A, B, C, was made with an Institute of Paper Chemistry brightness tester, commonly known as G. E. brightness tester, to determine the effect of hydrochloric acid and calcium chloride pretreatments at various wave lengths in the visible spectrum. First of all, the percent brightness as compared to the brightness of a freshly prepared block of magnesium carbonate was determined. The wave length was changed by inserting additional color filters. The wave lengths for these tests ranged from 400 to 651 millimicrons.

The percent brightness for all bleached pulps was determined with either Photovolt or G. E. testers as compared to glass standards.

PRESENTATION OF RESULTS

Several preliminary peroxide bleaches of chlorinated and caustic extracted pulp were made to determine the peroxide concentration necessary for a desired brightness. The results of these experiments can be seen in table I and figure 2.

A summary of all experiments performed may be seen in table II and figure 3. Active bleaching ingredients, pretreatments used, and percent brightness values are listed.

A color evaluation of the bleached pulp from experiment III - A, B, C, made to determine the effect on brightness of hydrochloric acid and calcium chloride pretreatments at various wave lengths in the visible spectrum, may be seen in table III and figure 4.

INTERPRETATION OF RESULTS

From the results of experiments I and II, as shown in table II and figure 3, it is evident that hydrochloric acid pretreatment of chlorinated and caustic extracted kraft pulp before last stage hydrogen peroxide bleaching is detrimental to brightness under the experimental conditions used. In experiment I the hydrochloric acid treated peroxide bleached pulp had a Photovolt brightness of 62.0 per cent while the untreated peroxide bleached pulp hade a brightness of 72.0 percent. This shows a decrease in brightness caused by acid pretreatment.

The results of experiment III and IV, as reported in table II and figure 3, also, show that hydrochloric acid pretreatment of chlorinated, caustic extracted and calcium hypochlorite bleached kraft pulp before peroxide bleaching is not beneficial to brightness under the conditions of these experiments. However, the amount of decrease in percent brightness due to acid pretreatment is not nearly as great in four stage bleaching as in a three stage system.

The calcium chloride pretreatment of kraft pulp, as reported in table II and figure 3, is beneficial to brightness under specific conditions. Experiments I and II show that calcium chloride treatment of chlorinated and caustic extracted kraft pulp before last stage peroxide bleaching resulted in an increase of two percent brightness as compared to the untreated standard pulp. The calcium chloride pretreatment of chlorinated, caustic extracted, and calcium hypochlorite bleached kraft pulp, however, resulted in approximately the same brightness as that of the untreated pulp. Therefore, under the conditions of these experiments calcium chloride pretreatment was beneficial only in three stage bleaching.

An evaluation of bleached pulp from experiment III as to reflectivity over the total range of the visible spectrum indicates that both hydrochloric acid and calcium chloride pretreatments result in an increase in brightness in the yellow-orange range of the spectrum. In the blue range of the spectrum the acid pretreatment is detrimental to brightness/and the calcium chloride pretreatment results in little change in brightness values. This means that the hydrochloric acid and calcium chloride treated pulp will appear more yellow than the untreated pulp. This condition is shown in table III and figure 4.

CONCLUSIONS

From the results of the experimental work conducted, it is concluded that hydrochloric acid pretreatment of chlorinated and caustic extracted kraft pulp before last stage peroxide bleaching appeared to be detrimental to final brightness. Also, hydrochloric acid pretreatment of chlorinated, caustic extracted, and calcium hypochlorite bleached kraft pulp before peroxide bleaching was not beneficial to final brightness, when two per cent available chlorine in the form of calcium hypochlorite was used as the third stage.

Calcium chloride pretreatment, on the other hand, was definitely of value to final brightness in three stage bleaching. However, calcium chloride pretreatment of chlorinated, caustic extracted, and calcium hypochlorite bleached kraft pulp did not increase final pulp brightness to a significant extent.

It seems, therfore, that there was an optimum concentration of calcium salts which was of maximum benefit to final brightness of last stage peroxide bleached kraft pulp.

Furthermore, there was some indication that both the hydrochloric acid and calcium chloride pretreatment of the calcium hypochlorite treated pulp caused small increases of reflectance in the yellow-orange range of the spectrum. Thus the pretreated pulp appeared to be more yellow than pulp bleached to the same brightness level without pretreatment.

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Table I. Preliminary Hydrogen Peroxide Bleach of Chlorinated and Caustic Extracted Pulp.

Percent H_2O_2 (50 %) based on weight of caustic extracted pulp	Percent Brightness (Photovolt)
0.0	30.0
1.0	53.0
3.0	63.0
6.0	70.5
8.0	71.0

Table II. Summary of Brightness Values.

Experiment number	% brightness after caustic extraction (Photovolt)	% active Cl_2 as calcium hypochlorite	% brightness after calcium hypochlorite	Treatment before bleaching with hydrogen peroxide	% H_2O_2 (50%) used based on pulp weight	Percent Brightness	
						Photovolt	G. E.
I-A	30.0	-	-	Hydrochloric acid	7.0	62.0	-
I-B	30.0	-	-	Calcium chloride	7.0	74.0	-
I-C	30.0	-	-	None	7.0	72.0	-
II-A	30.0	-	-	Hydrochloric acid	3.5	55.0	-
II-B	30.0	-	-	Calcium chloride	3.5	73.5	-
II-C	30.0	-	-	None	3.5	71.5	-
III-A	30.0	2.0	74.0	Hydrochloric acid	2.0	81.5	78.9
III-B	30.0	2.0	74.0	Calcium chloride	2.0	82.5	80.6
III-C	30.0	2.0	74.0	None	2.0	82.5	80.5
IV-A	30.0	2.0	73.5	Hydrochloric acid	1.0	79.0	78.5
IV-B	30.0	2.0	73.5	Calcium chloride	1.0	80.0	79.3
IV-C	30.0	2.0	73.5	None	1.0	79.2	79.6

Table III. Evaluation of Reflectivity over the Total Range of the Visible Spectrum.

Exp. no.	Color effect	violet	blue			green	yellow	orange		red
	Wave length (mu)	400	439	457	491	537	571	606	630	651
	Filter no.	2	3	1	4	5	6	7	8	9
III-A	Hydrochloric acid pretreatment	69.1	78.3	78.9	82.6	84.9	86.1	86.7	86.7	87.1
III-B	Calcium chloride pretreatment	70.8	78.1	80.6	82.9	84.6	85.8	86.4	86.3	86.2
III-C	No pretreatment	70.8	77.8	80.5	81.9	85.1	85.4	85.6	86.4	85.6

Figure 1. The Oxidation Potentials of Common Bleaching Agents. (17)

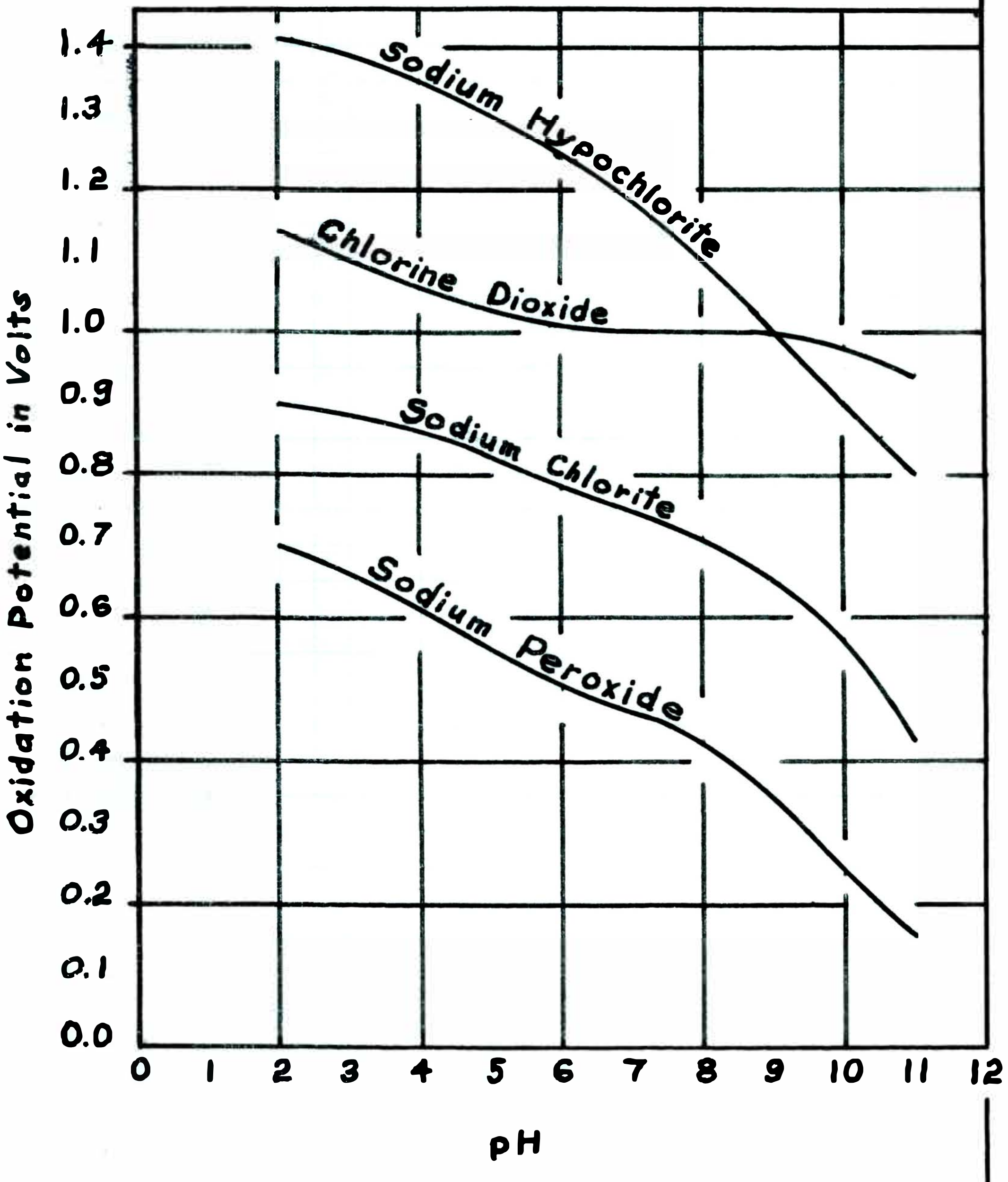

Figure 2. Brightness Evaluation of the Preliminary Hydrogen Peroxide Bleach of Chlorinated and Caustic Extracted Kraft Pulp.

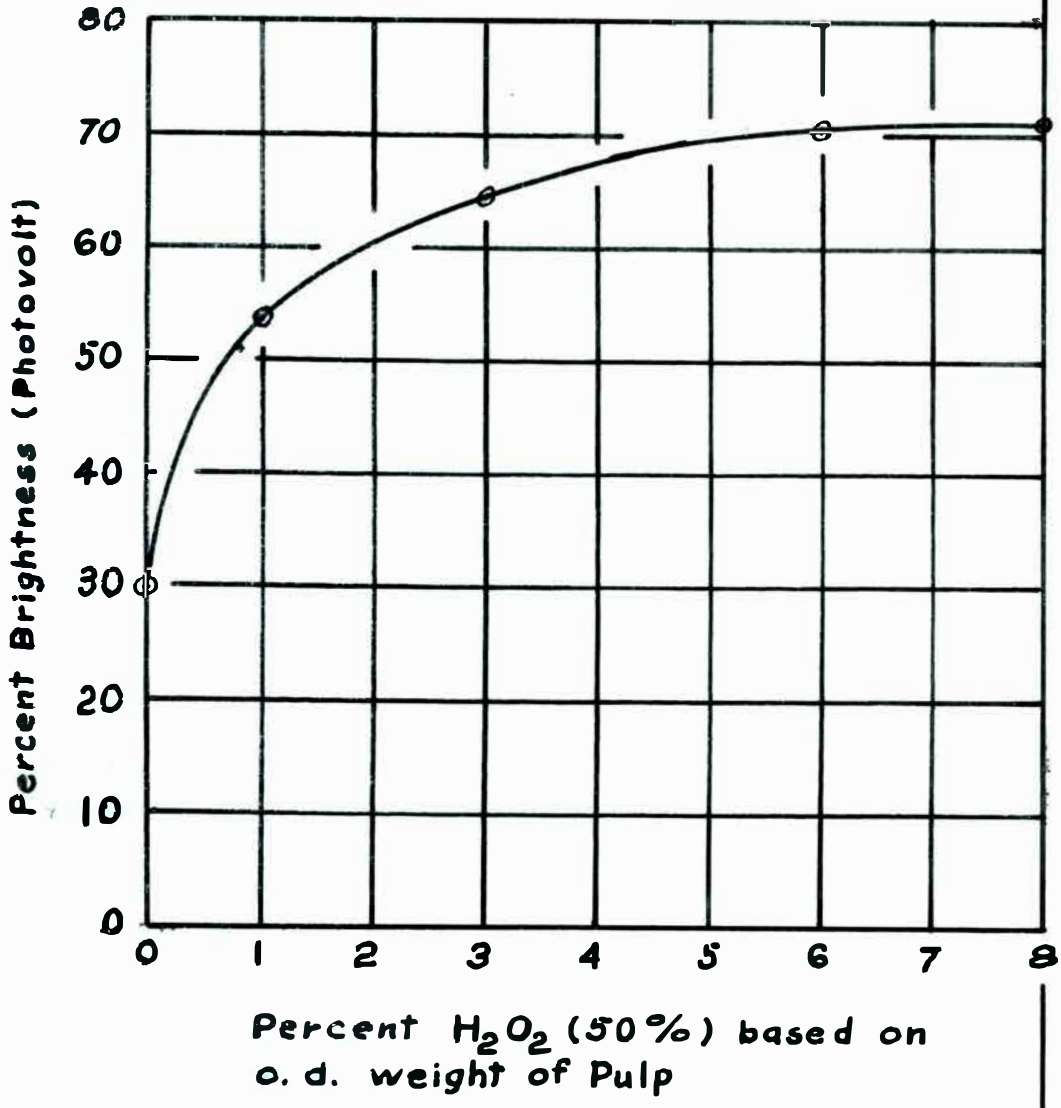

Figure 3. Summary of Brightness Values.

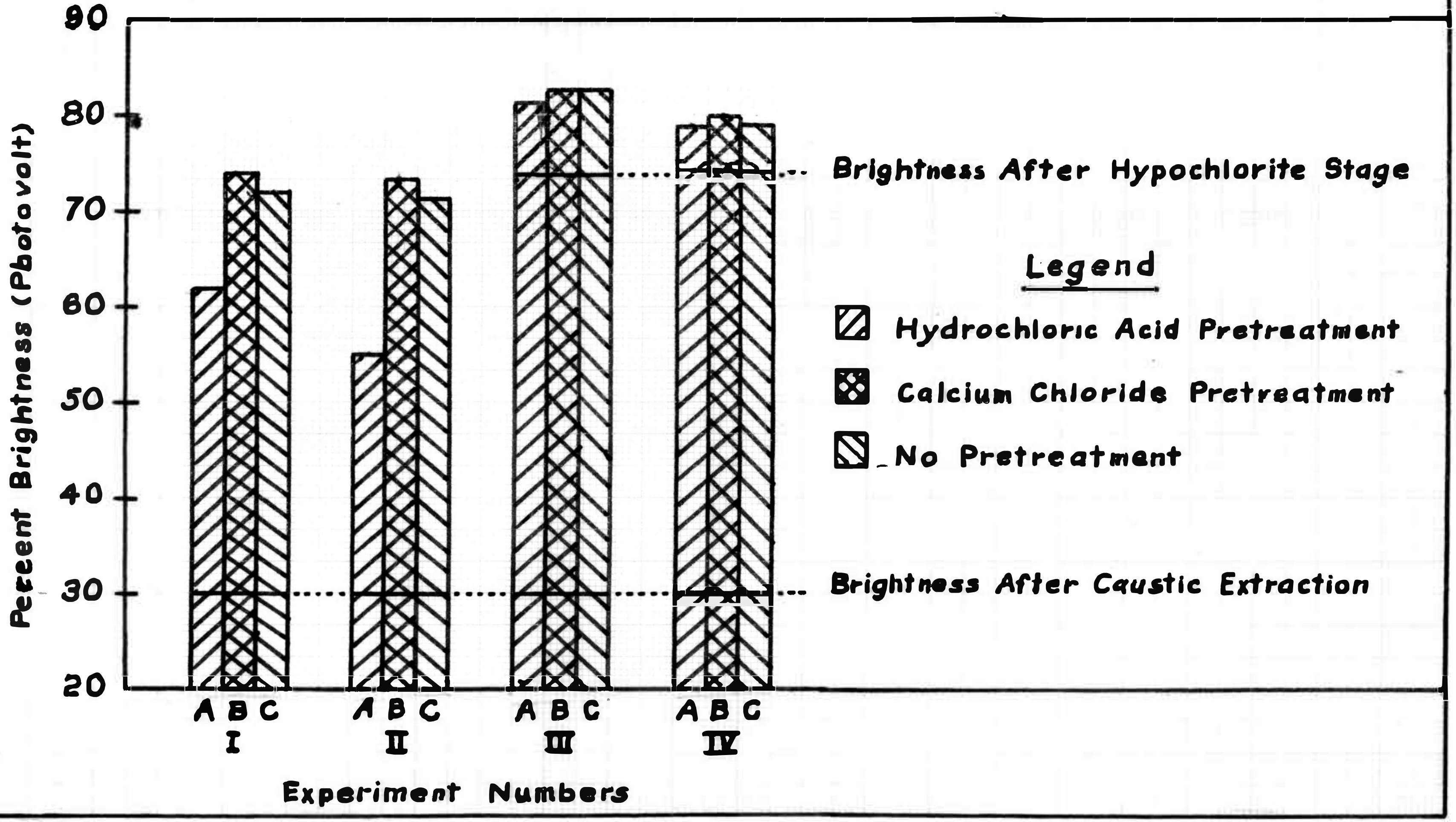

Figure 4. Evaluation of Reflectivity over the Total Range of the Visible Spectrum

Experiment III - A, B, C