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Research Report For GA Consortium

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Subject: Project Status Report

1. Project Title: Improved Peroxide-Based Bleaching Sequences For Georgia's Bleaching Operations - Part II

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3. Executive Summary of Work Completed:

Research studies for this past have accomplished most of the goals targeted for this project. The use of peroxide was to be studied on five kraft pulps including a commercial kraft pulp, AQ kraft, EMCC, AQ\polysulfide, and a cold blow. As reported on the following pages the bleaching studies for EMCC, commercial kraft and AQ are complete. The AQ/polysulfide pulp was recently prepared by Herty and is currently under study and the cold blow pulp studies are under progress. In addition to these studies the PIs have continued examining the bleachability of softwood kraft pulps with D/Z, Z/D, PHT. The bleaching conditions proposed for D/Z (0.3 & 0.5% charges) in the sequences D/Z(EOP)DED or D/Z(EOP)Z/D(EP)D did not afforded the target brightness values of +85. At this time we are uncertain if this was due to the presence of carry-over or insufficient ozone in the first stage. Additional experiments are be pursued to determine how the final brightness of this sequence can be improved. Currently we are also examining the brightness values for OZ(PHT)D and OZQ(PHT).

Summary
The lignin content of kraft pulp entering the bleach plant can be lowered through the use of extended delignification cooking technology or through the use of oxygen delignification. This work compares the bleachability of these two extended delignification processes as well as the conventional kraft process. The extended delignification technology used in this work was the Kraft-AQ process. The ECF bleach sequence employed in the comparison was DEopDD. Conventional kraft and kraft-AQ pulps studied had initial kappa numbers ranging from 15 to 30. Different overall bleach plant kappa factors were studied to determine the stoichiometry of bleaching for each of the cooking/delignification processes. The kraft-AQ cooking/delignification process gave us the best bleachability in terms of brightness, chemical requirement, and pulp viscosity.

INTRODUCTION

Due to increasing environmental regulatory pressure, chlorine in the first delignification stage of bleaching is being phased out. Many new technologies have been proposed to replace this very selective and cost-effective means of delignifying the kraft pulp. Of these technologies, the easiest to implement into the existing bleach plant is the complete replacement of chlorine with chlorine dioxide in the first bleaching stage. Chlorine dioxide is comparable to chlorine in its selectivity and delignification capability, but is far more expensive than chlorine. Because of this, it is widely recognized that the kappa number of the pulp entering the bleach plant must be lowered so that the bleaching chemical demand is lowered. Technologies that lower the kraft pulp kappa number include oxygen delignification, enhanced batch cooking technologies such as AQ and AQ/PS cooking, Rapid Displacement Heating extended batch cooking, and extended delignification in modified continuous digesters.

Technologies such as extended continuous cooking and RDH cooking can, however, affect the yield of the pulp. In this case, a tradeoff must be realized between the benefit of reducing the kappa number to use less bleaching chemicals, or in reducing the pulp yield. Extended cooking technologies such as AQ, AQ/PS, and kraft-O2 delignification can avoid the yield issue, however.

Most mills extend the cooking process using modified continuous cooking. In this process, the temperature profile and the effective alkali profile are leveled out in the continuous column. This provides a lower lignin content at higher viscosities than conventional cooking can provide. One drawback to this process is that the yield is often negatively impacted. Thus, in bleaching, final brightnesses comparable to conventional cooking can be obtained using less bleaching chemicals, but final pulp strength is typically no better than in bleaching of conventional kraft pulp.

Anthraquinone has long been known to provide an extended cook without the expense of yield loss1-4. Oxygen delignification coupled with higher kappa cooking can also provide a very low kappa number without appreciable yield loss6. The interruption of kraft pulping at a higher kappa followed by well-controlled oxygen delignification can give similar yields and kappa numbers as kraft-AQ pulp5. A comparison between the bleachability of high-kappa kraft-O2 pulp and kraft-AQ pulp is beyond the scope of this paper.

The purpose of this work is to compare the bleachability of conventional and extended delignified kraft pulp from the same wood source. An elemental chlorine free (ECF) bleaching process representative of many commercial operations is used to bleach the pulp. Conventional batch kraft and kraft-AQ pulps are compared in this work. The yield advantage of anthraquinone in pulping has long been known. The bleachability of AQ pulp compares favorably to conventional kraft pulp in a bleaching sequence containing chlorine in the first stage3,4. This work examines the bleachability in modern ECF sequences.

There is no general statement that can be made as to which process is the best in terms of pulp yield, bleached brightness, and bleaching cost, however. It is known that kraft-O2 pulps tend to require a higher bleach plant kappa factor than conventional or extended kraft pulp, but that kappa factor is based on inlet kappa number, so the overall amount of bleaching chemicals is lower7. The comparison between conventional kraft and kraft-AQ pulps is rather vague at this point. This work compares the bleaching response for conventional kraft and kraft-AQ pulps with identical starting kappa numbers.

EXPERIMENTAL

Kraft and kraft-AQ pulps were generated from Southern Pine chips at the Herty foundation. Several inlet kappa numbers and viscosities were used in this study, as illustrated in Table 1.

Pulp Type	Screened Kappa	Screened Yield	Viscosity, cp
Kraft 1	16.6	41.6	19.2
Kraft 2	29.2	45.5	31.8
Kraft-AQ 1	17.2	42.6	20.1
Kraft-AQ 2	26	46.5	28.4

Table 1. Properties of initial pulp supplied for bleachability comparison.

Please note that the standard kraft and the AQ kraft pulps were cooked to two different kappa levels, namely, ~30 and ~17. This was done so that a comparison could be made between the two differently cooked pulps that have roughly the same starting lignin content. The primary difference between the conventional kraft and the kraft-AQ pulps is the higher yield exhibited from the use of 0.1% AQ in the batch digester. The AQ addition provides for an extra 1% yield in all the cooks.

These pulps were each subjected to a D(PO)DND bleach sequence under the conditions found in Figure 1. All chlorine dioxide stages were performed in heat-sealable polyethylene bags immersed in a thermostatically controlled water bath. At the end of the D1 stage, or the second chlorine dioxide stage in the sequence, NaOH was charged into the pulp for 5 minutes to simulate neutralization in the dilution ring of the D1 tower. The NaOH charge was equal to half the active chlorine charge for that D stage. This then becomes what is known as a DN stage.

The enhanced oxidative extraction stages, or (PO) stages, for this portion of the work were carried out in a stainless steel reaction bomb kept at a constant temperature via a heating jacket. The pulp and water were preheated to 90°C in a water bath, and then the NaOH and H2O2 were mixed in by hand. The slurry was then transferred to the preheated reactor, the reactor was sealed, and oxygen gas was applied at 40 psig for 20 minutes. At the end of the 20 minutes, the oxygen pressure was released and the pulp was retained for an additional 45 minutes. This stage is oxidative extraction, but with the high charges of peroxide, 1% or 2%, they become known as (PO) stages.

In further work, an enhanced Eop stage is studied. The effect of split addition of peroxide was examined. For these experiments, the CRS 1015S reactor previously discussed9 was used. The pulp and water were first preheated to the desired temperature using the reactor controls. The bleach chemicals were injected using an injection device and oxygen pressure. Mixing was accomplished using the turbine at 1500 rpm for 10 seconds. In split addition experiments to be discussed later in this paper, part of the extraction chemicals were injected under pressure and mixing conditions at a specified time into the extraction stage.

Kappa numbers, brightnesses, and viscosities were all obtained using TAPPI standard methods.

RESULTS AND DISCUSSION

D(PO)DND Bleaching Sequence

Two levels of kappa number for kraft and kraft-AQ pulp were bleached in a D(PO)DND bleaching sequence at various levels of chemical application. Figure 1 shows the experimental conditions used. In effect, kraft pulp and kraft-AQ pulps had virtually identical kappa numbers going in to the bleaching experiments. Two levels of kappa factor were used in the Do stage, 0.15, and 0.25. For each of these pulps, both 1% and 2% H2O2 were charged on the Do pulp in a (PO) extraction stage. Then, the pulp from the 0.25 kappa factor D(PO) sequences were brought up to final brightness using various levels of ClO2 in a DND brightening sequence.

The results for these ECF bleaching sequences can be found in Figures 3 and 4. Figure 3 shows the brightness, kappa, and viscosity results for each bleaching operation for the conventional kraft pulp, and Figure 4a shows the same data for the kraft-AQ pulp. In comparing the kraft to the kraft-AQ pulp, Table 2 shows the final data.

Pulp Type	Final Brightness	Final Viscosity
kraft, 29 kappa	86	11.1
kraft, 16.6 kappa	86	7.7
kraft-AQ, 26 kappa	86	12.7
kraft-AQ, 17 kappa	85	8.5

Table 2. Final bleaching results for ECF bleaching. This table refers to kappa factor 0.25, 1% H2O2 in (PO) stage

Table 2 refers to the bleaching results which gave the highest brightness at acceptable viscosity levels, namely, 0.25 kappa factor in Do delignification and 1% peroxide charge in the (PO) extraction stage. The best overall bleaching result in terms of final brightness and viscosity was for the kraft-AQ pulp with a 26 initial kappa number. This pulp had a final brightness that was four points higher than its counterpart kraft pulp with the 29 starting kappa number, although the final viscosities were the same. This indicates that the use of AQ to pulp to higher kappa numbers makes the pulp more bleachable in the ECF bleaching process.

Anthraquinone in kraft pulping allows lower kappa numbers to be reached at identical pulp yields1-4, or it allows higher yields at identical kappa numbers. Therefore, we would expect the bleached pulp from the low kappa kraft-AQ pulp to have a higher brightness and higher strength than the low kappa conventional kraft pulp. As Table 1 and Figures 3 and 4 show, although the final brightness was 1 point lower for the kraft-AQ, the viscosity was 2 cp's higher, indicating better bleaching selectivity. Thus, the AQ addition to the kraft process generated pulp that has better bleachability to ECF bleaching processes.

One of the interesting results of this study was that the brightness gain was relatively low, and the viscosity loss was fairly high for the (PO) extraction stages. Table 3 shows representative data for the two types of pulp, based on the same bleaching results given in Table 2.

The AQ pulps gave slightly higher brightnesses after the (PO) stage, and they also gave higher viscosities. This indicates that AQ pulp is more selective to potentially harmful bleaching processes. For all these D(PO) processes, virtually no metal management procedures were followed. The initial pH in the all Do stages was 3.5 - 4.0, which is necessary for AOX abatement8. This pH is a bit too high for effective transition metal removal in an acidic ClO2 stage. If the pH was lower, metals would be washed from the pulp, but this is not good from an operational standpoint. Further metal removal procedures would include the use of a (D+Q) stage, where DTPA or EDTA is introduced at the end of the Do stageREF.

Pulp Type	D(PO) Kappa	D(PO) Brightness	D(PO) Viscosity
Kraft 29	3.3	61	13.6
Kraft 16.6	2.1	61	7.9
Kraft-AQ 26	3.6	62	14.6
Kraft-AQ 17	2	63	10.7

Table 3. D(PO) bleaching summary for comparing kraft to kraft-AQ pulp. Do kappa factor = 0.25 and peroxide charge in (PO) = 1% for these runs.

Typical peroxide-enhanced extraction stages only use 0.2 - 0.5% H2O2 charge. In this study, 1 to 2% peroxide was charged, which will dramatically increase the rate of peroxide decomposition9. This caused the brightness and viscosity after the (PO) stage to be lower than expected. The use of AQ in the pulping process, however, led to better selectivity of the pulp in the damaging (PO) stage in all cases, however. Thus, when combined with a proper metal management step, AQ pulp should have even better final brightness and viscosity than that reported here.

As expected, the pulps using the 0.25 kappa factor gave the highest final brightnesses. It is interesting to note that the final brightnesses were about the same for all the starting pulps. This means that, although AQ pulp has a higher selectivity, it does not tend to give higher brightnesses at similar starting kappa numbers to conventional kraft pulp. The key to higher brightness with identical chemical charge, then, is related to the starting kappa number rather than the amount of cellulose in the pulp. The reduction in total chlorine dioxide charge to reach final brightness by lowering the starting kappa number was 28% for conventional kraft and 28% for kraft-AQ. This means that both pulps have the same potential for chemical savings at lower starting kappa numbers, but the AQ pulp gives a slightly higher viscosity.

A(PO)DED Bleaching Sequence

The same levels of kappa number for kraft and kraft-AQ pulp were bleached in a A(PO)DED sequence as were bleached in the earlier sequence. This was done to introduce a metal management stage to the ECF sequence. In this sequence, the workhorse of delignification is the (PO) stage. Delignification across the A(PO) sequence met or exceeded 50% in all the cases. Figure 2 shows the experimental conditions used. Again, the kappa numbers at the beginning of the bleach sequence were virtually identical. The pH was 2.5 at 90C for 60 minutes. Each pulp was then subjected to 1 % and 2% H2O2 during the (PO) extraction stage. After this, the same two kappa levels were used as in the earlier bleaching sequence. The pulp from the 0.25 kappa factor were furthered brightened using three levels of ClO2 in a DN stage.

Figures 5 and 6 show the results from this ECF bleaching sequences. These figures report the kappa number, brightness, and viscosity after various conditions. Table 4 compares the final results for kraft and kraft-AQ.

Pulp Type	Final Brightness	Final Viscosity
kraft, 29 kappa	70	9.8
kraft, 16.6 kappa	74	6.7
kraft-AQ, 26 kappa	67	8.9
kraft-AQ, 17 kappa	77	5.6

Table 4. Final bleaching results for ECF bleaching. This refers to kappa factor 0.25, 2% H2O2 in (PO) stage.

Table 4 shows the results that gave the highest brightness after the D0 stage. This corresponded to 0.25 kappa factor and 1% H2O2. The kraft-AQ with a 17 initial kappa number gave the highest final brightness. These results indicate that the acid wash and (PO) stage before the D0 stage was not effective.

It is interesting to note that the high kappa number kraft pulp performed better than the high kappa number kraft-AQ pulp. The low kappa number kraft-AQ, however, outperformed the corresponding kraft pulp. These results are completely opposite from those of the other bleaching sequence.

SPLIT PEROXIDE ADDITION IN (PO)

We attempted to enhance the (PO) stage through split addition of peroxide in the pressurized portion of the (PO) stage. As discussed in the Experimental section of this paper, the split addition (PO) stages were run at 110°C for 30 minutes at 70 psig oxygen pressure, and for 60 additional minutes at 90°C, atmospheric pressure. Peroxide was injected either all at the beginning with the NaOH, half at the beginning and half after 10 minutes reaction time, or all after 10 minutes reaction time. The theory behind using a split peroxide addition is that the peroxide will react with the extracted filtrate preferentially to the residual lignin in the pulp. If the peroxide charge is delayed somewhat, the extraction mechanism can go to completion, then the peroxide can do its job of brightening and delignifying the pulp. It has been shown10 that peroxide reacts better in two stages if no intermediate washing is done between the stages than if there is intermediate washing between the stages. This concept was then applied in a split peroxide addition (PO) extraction stage. Table 5 shows the final results of the split addition experiments.

Type of Addition	D(PO) Brightness	D(PO) Viscosity	(PO) Kappa
All peroxide at start	52.2	11.9	5.2
½ at start, ½ after 10 min	53.0	10.9	4.3
all after 10 min	51.1	11.6	4.4

Table 5. Post (PO) stage results for split addition peroxide in D(PO) delignification sequence. Pulp initial kappa number was 21.3, viscosity was 22.8 cp.

The kappa factor for the experiments in Table 5 was 0.2, and a conventional kraft pulp was used. It can be seen that the split addition where half the peroxide was charged at the beginning and half after 10 minutes gave slight improvements in post (PO) stage brightness. These results carried through to the final brightnesses as well. The experiment where all the peroxide is charged after 10 minutes gave a lower brightness at similar kappa reduction, indicating a reaction between the peroxide and the extracted lignin in the bleach liquor instead of with the pulp itself. By charging some initial peroxide and then waiting to charge the rest, the initial extracted lignin reaction with the peroxide is complete by the time the remainder of the peroxide is charged into the reactor.

CONCLUSIONS

ECF bleachability for kraft-AQ pulp is similar in brightening response than for conventional kraft pulp. The selectivity of kraft-AQ pulp, however, is better than for conventional kraft pulp. Additionally, kraft-AQ pulp has much better selectivity in a cellulose-harmful environment such as a (PO) stage without good metal management. In all cases, the high-kappa pulps perform better selectivity than the low kappa pulps, although they require more chlorine dioxide to reach high brightness. In future work, the effects of better metal management in these bleaching processes will be examined.

The use of a split peroxide addition (PO) or Eop process may prove beneficial to final brightening in an ECF process. The initial charge of peroxide reacts with extracted lignin so that the remainder of the peroxide can react with the residual lignin still in the pulp after some reaction time has elapsed.

Improved Peroxide-Based Bleaching Sequences for Georgia's Kraft Bleaching Operations- Pt.II

Pulping of Southern Pine using Commercial Kraft, Polysulfide, and AQ Pulping Processes

For

Georgia Consortium For Technological Competitiveness in Pulp And Paper

Final Report For FY 1997

September 23, 1997

Herty Project Number 710147

Herty Project Managers: Lorenzo Bryant / Martha Simmons

Herty Foundation
Savannah, Georgia

I. Objective

Prepare a series of low-kappa pulps employing advanced kraft pulping technologies and pulping additives such as AQ and polysulfide. EMCC and RDH cold blow pulps were commercially produced at Southeastern mill operations.

II. Summary of Project

The pulps produced at Herty and the commercially obtained pulps were manufactured from regionally available Southern pine species. The pulps obtained and produced at Herty were representative of new kraft pulping technologies which are viable for future kraft pulping operations in this state.

A total of 10 cooks were made in the laboratory.

The target kappas for the commercial kraft pulps were 30 ± 2, 25 ± 2 and 20 ± 2.

The target kappas for the AQ kraft pulps were 30 ± 2, 25 ± 2 and 20 ± 20.

The target kappas for the polysulfide sulfide pulps were 25 ± 2, 20 ± 2 and 15 ± 2.

The cooks produced at Herty were evaluated for total yield, screened yield, and rejects.

All pulps were evaluated for kappa number (TAPPI T236) and capillary viscosity (TAPPI T230) prior to shipment to Georgia Institute of Technology and the Institute of Paper Science and Technology.

III. Materials and Procedures

A. Materials Supplied By Client:

1. None

B. Materials Supplied by Herty:

1. Southern Pine Chips obtained from a local mill
2. Kraft White liquor
3. Polysulfide Liquor
4. Kraft AQ Liquor

C. Chip Preparation and Classifications

The locally obtained chips were screened by thickness and length at the mill. The chips were thoroughly mixed, randomly sampled for moisture determinations, and stored in sealed drums.

After obtaining moisture determinations, the chips were bagged in polyethylene bags based on the amount of chips necessary for each cook and then stored at 40°F prior to pulping.

D. Cooking Liquor

The commercial kraft white liquor was prepared in the laboratory using commercially purchased sodium hydroxide and sodium sulfide.

The polysulfide liquor was prepared by dissolving approximately 250 grams of sulfur powder in 2 liters of sodium sulfide aqueous solution (120 g/L) at 60°C. A mixture if the white liquor, polysulfide liquor, NaOH solution, and water was prepared as the cooking liquor.

For cooks requiring anthraquinone, the required amount of anthraquinone was thoroughly mixed with the cooking liquor before adding to the chips in the digester.

The liquors were titrated for active alkali, effective alkali, and sulfidity according to an industry accepted ABC titration method.

E. Pulping Of Low Kappa Pulps:

Table I contains the pulping conditions for each cook performed.

A 0.78 cu. ft. laboratory digester was preheated to 212°F prior to each cook. 2000 grams (ovendry weight) of chips were added to the preheated digester and sufficient white liquor (necessary to obtain the required percentage of effective alkali on ovendry wood) and water (necessary for obtaining a 3.5:1 ratio of liquid-to-wood) were added. The liquor and water mixture was continuously circulated during the entire cooking cycle. The cooks were made using indirect steam.

At the end of the cooking cycle, the cooks were blown at 90 psig and subsequently washed with room temperature tap water until wash water was clear.

F. Pulp Testing Of Low Kappa Pulps:

The washed pulps were dewatered and weighed for total yield determinations (Table I).

The pulps were screened on a 6-cut laboratory flat screen for determining screened kappa number, screened yield and percent rejects determinations. (Table I).

Table I also contains the residual liquor analyses.

Table II contains the kappa number and viscosity data on the EMCC and commercial RDH cold blow pulps.

IV. Equipment Used

1. Cold room for chip storage, 8 feet x 12 feet , maintained at 40°F.

2. Vertical Otto - Biefield Co. Digester, 304 ss, 0.78 cu.ft. capacity, 120 psi, indirect or direct heat, liquor circulation pump, ss blow tank, 3:1 liquor-to-wood ratio minimum.

3. Sprout- Waldron laboratory flat screen, 12 inches x 43 inches with a 6-cut screen.

4. Wine press for dewatering pulp.

5. Standard analytical laboratory equipment for white and black liquor titrations, kappa numbers, and scales for weighing pulps for yield determinations.

Project 710147

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Table I
Pulping Data Summary

COOK NO.	1 Commercial Kraft	2 Commercial Kraft	3 Commercial Kraft	4 Commercial Kraft	5 Kraft AQ	6 Kraft AQ	7 Kraft AQ	8 AQ/ Polysulfide	9 AQ/ Polysulfide	10 AQ/ Polysulfide
CHIP FURNISH	Pine	Pine	Pine	Pine	Pine	Pine	Pine	Pine	Pine	Pine
CONDITIONS:										
Chip Moisture %	51.85	49.20	51.85	51.85	49.20	49.20	49.20	49.20	49.20	49.20
Liquor:Wood Ratio	4:1	4:1	4:1	4:1	4:1	4:1	4:1	4:1	4:1	4:1
Chip Charge, (ovendry) grams	2000	2000	2000	2000	2000	2000	2000	2000	2000	2000
Total Liquid, mL	8000	8000	8000	8000	8000	8000	8000	8000	8000	8000
Time Up, min.	90	90	90	90	90	90	90	90	90	90
Time At, min.	110	105	70	140	105	90	75	125	110	98
Temperature, °F	340	340	340	340	340	340	340	340	340	340
H-Factor	2212	2127	1553	2652	2301	1864	1620	2489	2290	2082
Chemicals:										
Active Alkali as Na_20 on ovendry wood, %	24.0		19.5	24.0		19.5	19.5			
Effective Alkali as Na_20 on ovendry wood, %	21.0	18.0	17.0	21.0	18.0	17.0	17.0	20.0	18.0	16.0
Sulfidity as Na_20, %	25.0	25.0	25.0	25.0	25.0	25.0	25.0	*	*	*
Pulping Additives, % on ovendry wood										
A.Q. %	0	0	0	0	0.1	0.1	0.1	0.1	0.1	0.1
Residual Liquor:										
Effective alkali as Na_20, g/l	17.36	15.97	15.50	19.53	16.12	13.80	13.95	---	---	---
Active alkali as Na_20, g/l	27.44	25.89	25.11	29.14	25.73	23.89	22.94	---	---	---
Pulp results:										
Total Yield, %	43.23	46.79	47.88	39.68	43.76	47.10	48.26	37.78	42.88	44.37
Screened Yield, %	41.61	44.87	45.53	38.74	42.58	45.61	46.51	37.09	41.82	42.99
Rejects, % (6 Cut Flat Screen)	3.74	4.11	4.86	2.37	2.67	3.17	3.62	1.83	2.45	3.13
Screened Kappa Number	20.53	24.84	32.40	15.65	20.67	25.13	29.62	15.43	18.0	24.5
Viscosity	19.22	22.76	31.82	13.14	20.14	24.94	28.40	14.29	20.60	24.82

* Actual sulfidity could not be titrated but based on information received from client, sulfidity should be greater than 28%.

TABLE II

Project 710147

RDH COLD BLOW AND EMCC PULPS

SAMPLE IDENTIFICATION	Commercial RDH Cold Blow Pulp*	EMCC**
Kappa Number, T236	25.39	12.3
CED Viscosity, T230	22.25	21.0

* Pulp source: Williamette Industries; Bennettsville, SC
  Southern Pine

** Pulp source: Stone Savannah River Mill, Savannah, GA