

U.S. DEPARTMENT OF ENERGY  
FEDERAL ASSISTANCE PROGRAM/PROJECT STATUS REPORT

OMB Burden Disclosure Statement

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1. Program/Project Identification No. <b>DE-FC07-96ID13442</b>	2. Program/Project Title <b>High-Efficiency Chlorine Dioxide Delignification</b>	3. Reporting Period <b>4/1/98 through 6/30/98</b>
4. Name and Address <b>Thomas J. McDonough 500 10th Street, NW, Atlanta, GA 30318 (404) 894 - 97017, thomas.mcdonough@ipst.edu</b>		5. Program/Project Start Date <b>8/9/96</b>
		6. Completion Date <b>Extended 1 yr to 7/9/99</b>

7. Approach Changes  <input checked="" type="checkbox"/> None	<b>DOE/ID/13442--71</b>
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8. Performance Variances, Accomplishments, or Problems <ul style="list-style-type: none"><li>Comparative conventional D<sub>0</sub>(EO) bleaching of pulps from conventional and modified processes showed slightly improved bleachability for the latter and confirmed high efficiencies and low chlorate yields at low kappa factor</li><li>Effectiveness of ultralow-AOX ClO<sub>2</sub> delignification process demonstrated</li><li>Process for high-efficiency delignification identified</li><li>Completed experiments to study mechanism by which low kappa factors give high efficiency</li><li>Determined relationship between hexeneuronic acid content and unbleached kappa number of hardwood pulps</li><li>Presented paper at 1998 International Pulp Bleaching Conference</li></ul> <input type="checkbox"/> None
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9. Open Items <ul style="list-style-type: none"><li>Molecular weight analysis of softwood kraft continues</li><li>Isolation and analysis of conventional and vapor phase bleached, unbleached hardwood pulp residual lignins continuing</li><li>Analysis of data from low-kappa-factor efficiency study underway</li><li>Further development of high-efficiency delignification process in progress</li></ul> <input type="checkbox"/> None
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10. Status Assessment and Forecast <ul style="list-style-type: none"><li>Project behind schedule by 6+ months due to Olympics related startup delay and loss of postdoctoral research fellow. Replacement PDF now fully productive and current progress in NMR and molecular weight studies is good. One co-p.i. (AJR) currently on 6-month sabbatical in Sweden. One-year no-cost extension was requested and has been granted.</li></ul> <input type="checkbox"/> No Deviation from Plan is Expected
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11. Description of Attachments <ul style="list-style-type: none"><li>Brief summary of results obtained during the second quarter of 1998 is attached.</li></ul> <input type="checkbox"/> None
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12. Signature of Recipient and Date <i>Thomas J. McDonough</i> July 31, 1998 Thomas J. McDonough, July 31, 1998	13. Signature of U.S. Department of Energy (DOE) Reviewing Representative and Date
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## High-Efficiency Chlorine Dioxide Delignification

U.S. Dept. of Energy Project DE-FC07-96ID13442

### Summary of Progress

April 1 - June 30, 1998

Arthur J. Ragauskas and Thomas J. McDonough  
Institute of Paper Science and Technology

We have been pursuing strategies for improving the efficiency of utilization and environmental performance of  $\text{ClO}_2$  in the delignification of kraft pulps. These strategies will apply several of the findings of this project. These strategies combine a pretreatment followed by  $\text{ClO}_2$  treatment under conditions selected to result in high efficiency, and a suitably altered alkaline extraction stage. The potential for patentability of these strategies is currently being investigated. Detailed results will be presented after we have made a determination of their patentability.

In addition, a series of experiments was completed in which we compared the response to conventional D(EO) delignification of conventional kraft pulps and pulps from a laboratory simulation of Ahlstrom's Lo-Solids<sup>®</sup> extended delignification (modified) process. These experiments were conducted over a range of kappa numbers for both pulping processes, and at high and low kappa factors. The results (Table 1) showed that the modified pulps were slightly easier to bleach in the conventional process, an observation which parallels the corresponding observation made earlier, when the same pulps were bleached with the "Rapid  $\text{D}_0$ " short-retention process. As in previous work, there was a slight deterioration in bleachability as the unbleached kappa number was decreased. The bleachabilities of these pulps will be correlated with the structural features of the residual lignins they contain, to add to our database of lignin structure-bleachability relationships.

In other experiments, two sets of hardwood pulps of varied unbleached kappa number were subjected to determinations of their hexenuronic acid contents. Pulps representing both conventional batch and extended (RDH) kraft pulping processes were included. Hexenuronic acids are carbohydrate-derived components formed during kraft pulping by demethoxylation of a hemicellulose, 4-O-methyl-D-glucuronoxylan ("xylan"). Their presence in bleachable grade pulps is significant for two reasons: (1) they falsely inflate the unbleached kappa number, and (2) they consume electrophilic bleaching chemicals, such as chlorine dioxide. The analyses we performed consisted of acid hydrolysis under standard conditions, followed by spectrophotometric determination of the hydrolysis product (2-furoic acid) in the hydrolyzates. An alternate method is to determine the kappa number reduction that accompanies hydrolysis. The results we obtained are shown in Figure 1. It is apparent from the figure that, at a given kappa number, the two pulp types did not differ with respect to their hexenuronic acid contents. On the other hand, there was a significant effect of unbleached kappa number. As the kappa number was decreased below 30 by extending the kraft cook, the hexenuronic acid content first increased, then passed through a maximum, following which it decreased sharply. This finding will have considerable practical implication in the manufacture of hardwood kraft pulps for bleachable grades.

Residual lignin and effluent structural analyses by NMR and gel permeation chromatography are continuing to generate data for the development of structure-bleachability relationships. Analysis of this data is in progress.

Table 1. Conventional D(EO) Bleaching Parameters

Pulp Type	Kappa No.	Bright-ness	D <sub>0</sub> Stage						(EO) Stage				Total				
			Kappa Factor	Kappa No.	Delta Kappa/ TAC	AOX Yield, %	Cl <sup>-</sup> Yield, %	ClO <sub>3</sub> <sup>-</sup> Yield, %	Kappa No.	Bright-ness	AOX Yield, %	Cl <sup>-</sup> Yield, %	Delta Kappa/ TAC	AOX Yield, %	Cl <sup>-</sup> Yield, %	ClO <sub>3</sub> <sup>-</sup> Yield, %	Cl Recovery, %
Conv.	29.6	26.0	0.05	25.0	3.1	5.4	(13)	1	15.3	36.1	3.5	12	9.7	8.9	25	1	35
			0.20	14.8	2.5	8.8	48	15	5.7	49.2	2.6	10	4.0	11.5	58	15	84
	21.3	28.9	0.20	10.5	2.5	8.3	51	16	4.1	52.1	2.6	10	4.0	10.9	61	16	89
Lo-Solids	14.9	29.1	0.20	7.6	2.4	7.0	55	15	3.2	54.3	2.4	9	3.9	9.4	64	15	89
	28.7	25.2	0.05	24.9	2.6	4.9	54	5	14.3	35.1	3.5	13	10.0	15.3	67	5	81
			0.20	14.5	2.5	8.5	52	17	4.7	49.2	0.9	8	4.2	9.3	60	17	87
	18.8	28.1	0.20	9.4	2.5	8.5	52	15	3.2	54.3	2.7	10	4.1	11.2	62	15	89
	15.6	28.8	0.20	7.9	2.5	7.2	52	14	3	55.5	2.3	10	4.0	9.5	62	14	86

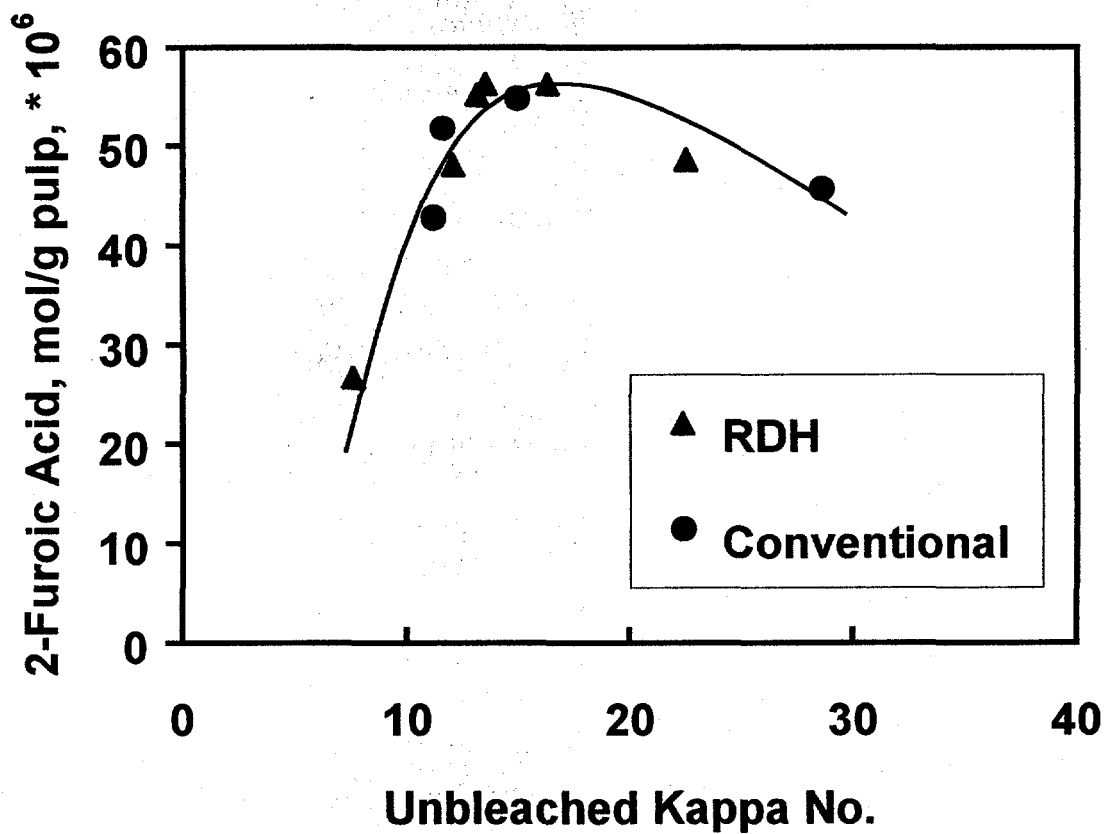


Figure 1. Hexeneuronic acid content of unbleached RDH and conventional kraft pulps from sweetgum.