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FY 97 PROGRESS REPORT
(01-JUL-96 THROUGH 30-JUN-97)

August 15, 1997

1. **PROJECT TITLE:** **Reducing Fresh Water Consumption in Papermaking**

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3. **PREFACE:**

Objectives and Goals:

The objective of this research is to enable Georgia paper mills to reduce their use of fresh water in the papermaking process by developing the capability to model and control the concentration build-up of nonprocess elements and other contaminants in paper machine white water systems. Two project goals have been established to meet this objective:

Goal A. Develop a validated computer simulation "module" for the general prediction of white water chemical equilibrium composition.

Goal B. Identify and evaluate one or more potentially viable purge technologies.

More than 90% of the FY97 funding ($103,885) was dedicated to achieving Goal A. Work towards Goal A is currently behind schedule. The reason for the slow progress is that a key associate investigator, Dr. P.S. Bryant, left IPST immediately prior to the start of the project and we were unable to replace his expertise until the middle of FY97. In addition, progress in the second half of FY97 was hampered by difficulty in securing mill participation (see Deliverable 1 below). Consequently, at the close of FY97, a budget balance of approximately $80,000 remained. A four month no cost time extension was approved to carry that amount forward until October 31, 1997.

Work towards Goal B is funded largely by matching funds. Progress in this area is on schedule and within budget. The original layout of the NPE Purge Evaluation Tasks in the FY97 Proposal Time Table (see **Attachment I**) has proven to be somewhat misleading. The first task listed is Laboratory Experiments in Precipitation (to be conducted 01-JUL-96 through 31-DEC-97). The third task listed is Development of Precipitation Purge (01-JUL-97 through 31-DEC-98). The implication and original intent here was to use the laboratory findings to develop an NPE purge technology based on precipitation. In reality, the precipitation experiments are intended to build an understanding of the impact of precipitation on the effectiveness of an electrodialysis-based purge technology. The Development of Precipitation Purge task (NPE Purge Evaluation Task No. 3) will not be pursued unless it can first be established by literature review and preliminary experimentation that precipitation is a potentially viable approach to purging NPE's from paper machine white water systems.

Difficulties Encountered and Options Considered:

Difficulties have been encountered in identifying a Georgia pulp and paper mill that is willing to participate in the project. Two mills that have been approached (i.e., Union Camp, Savannah and Georgia-Pacific, Brunswick) have elected to decline participation, citing a variety of reasons. Generally, mill interest levels and resources are low right now. We therefore re-evaluated our original project plan and identified options that could be pursued to accelerate the project's pace.

Options with the potential to speed progress:

1. Have IPST exert more pressure on mills to get involved.
2. Have Corporate level personnel exert more pressure.
3. Keep searching among other mills for one willing to participate.
4. Revise plan so that immediate progress is not impeded.

Exerting external pressure on disinterested mills will not work because we don't want to end up trying to conduct a sampling program in a mill that doesn't want us there. Continuing to search for a willing participant is viable and will be pursued. However, the process is time-consuming and we cannot afford to fall further behind in the project. Thus, it became imperative that we revise the project plan so that immediate progress could be made.

4. PROJECT PLAN REVISION:

Several planning meetings have been held in recent weeks between IPST Faculty, Senior Research Staff, and a Student to re-assess the overall project plan. General highlights of an agreed-upon revised plan are listed below. The newly revised plan does not alter the scope of the project in any way. It merely changes the order in which defined tasks are performed.

Revised Plan Highlights:

1. De-emphasize mill involvement, paper machine simulation development, and sampling program at this time.
2. Focus immediate efforts instead on the development of a preliminary mathematical model that will predict NPE (nonprocess element) behavior in model white water systems. NPE behavior in this context is defined as the physical partitioning of NPE's between aqueous solid (dissolved), non-pulp suspended solid (precipitated), and pulp (or other solid)-bound phases.

3. Collaborate with G. Jones of Simons Technologies, Inc. to review closely related internal IPST study.
   a) Identify appropriate base line white water and feed stock compositions that can be used as starting points in the statistical design of laboratory experiments.
   b) Re-assess the viability of IPST's tentative mill white water sampling and analysis plan.
4. Statistically design laboratory experiments aimed at understanding the behavior of NPE's under various model white water compositions and physical conditions.
5. Conduct laboratory experiments.
   a) Acquire Technician (Temp.) by 02-SEP-97; Post-Doc by 15-SEP-97.
6. Use laboratory data to develop/validate computational methodology that predicts NPE behavior in model white water systems. This will likely involve some type of interfacing (i.e., data exchange) with OLI solution equilibrium software, a task that will require assistance from Simons and/or OLI.
7. Conduct a minimal mill sampling program, characterize samples, and perform a preliminary evaluation of the predictive performance of the laboratory-generated mathematical model with real mill samples.

This plan will quickly get us back on schedule with respect to the laboratory experimentation phase of the project and will put us well ahead of schedule with the mathematical modeling task.

As a working model begins to take shape, we will have something more tangible with which to attract mill interest and mill participation should be easier to win over. A more comprehensive mill sampling plan can then be conducted at one (or maybe more) mill(s) to validate and tune the predictive model. Collaboration will again pick up with Simons at this point as we work through the problems of interfacing between the following possible realms: IDEAS™ Objects, user defined functions (UDF's), spreadsheets, and the OLI solution equilibrium software.

Once we have in-hand a <u>validated</u> NPE predictive model, we will turn over the computational methodology to Simons Technologies, Inc. and Pacific Simulation, Inc. so that it can be converted into a format that is compatible with each of their respective source codes. The final deliverable, and ultimate objective of this project, will be a switchable "option" residing in a WinGEMS™ Module and an IDEAS™ Object that will predict NPE behavior under a variety of paper machine white water conditions (physical and compositional).

The development of one or more paper machine wet end simulation model(s), originally the first task assigned in this project, now becomes one of the last tasks (i.e., scheduled for late FY98 and beyond, if funding continues through FY99) because it really is not essential to the development of the mathematical model. The purpose of the wet end simulation model(s) is to provide estimates of unavailable flow data so that the NPE model can be more completely tuned and validated. In addition, the wet end simulation model(s) will provide, to any participating mill(s), a tool that will immediately be able to utilize the final deliverable (WinGEMS™ Module/IDEAS™ Object "option") for the "what-if" evaluation of various mill closure scenarios. Non-participating mills can benefit from the final deliverable by merely compiling their own wet end simulation model, a task that is relatively straightforward and routine.

4. EXECUTIVE SUMMARY OF WORK COMPLETED:

NPE Simulation Tasks

Deliverable 1: Conventional simulation of paper machine wet end(s).

As mentioned above (see **Preface**), difficulties in attracting a willing mill participant have prompted us to put a lower priority on this task. This task is not critical to the NPE model development and its re-scheduling will have no impact on the final outcome of the project. The only reason it was originally scheduled at the front of the project was because that's the way it was done in other closely related NPE modeling work.

We are in the process of negotiating a subcontract agreement with Simons Technologies, Inc. that will call for them to provide consulting to assist IPST in the performance of this task. Their expertise in using the IDEAS™ software for process simulation will not only speed the completion of this task but will also ensure that the resultant wet end simulation(s) meets the highest professional engineering standards. This subcontract agreement is coupled with a software licensing agreement that allows IPST to use the IDEAS™ software at no cost for a period of 18 months.

Deliverable 2: Baseline sampling and testing.

A detailed tentative plan for this task is complete. **Figure 1** shows a schematic outlining the planned handling and analysis of the white water and feedstock samples. The above-mentioned collaboration with Simons will include a review of a similar mill sampling effort and should allow us to avoid pitfalls that are not immediately obvious in the tentative plan (see **Revised Plan Highlights,** Item 3b). The identification of specific sampling points, a consideration of the influence of machine dynamics on sample representativeness, and a plan rehearsal are some of the prerequisites that must be met prior to plan finalization.

A total of 29 trace metals will be screened for, as well as several inorganic anionic species of interest, including chloride, sulfate, carbonate, bicarbonate, and possibly others, depending on the type of wet-end additives in use at the participating mill. Several other conventional water analyses will be performed on raw samples in the field, including specific gravity, conductivity, temperature, and pH. Filtration will be performed on raw sample aliquots and alkalinity will be measured in the field by titration.

Other raw sample aliquots will be filtered on 0.40 micron polycarbonate membranes to separate the majority of suspended solids from the dissolved solids, thus allowing for the measurement of Total Dissolved Solids (TDS) and Total Suspended Solids (TSS). From these two determinations, Total Solids (TS) will be calculated and the result checked for

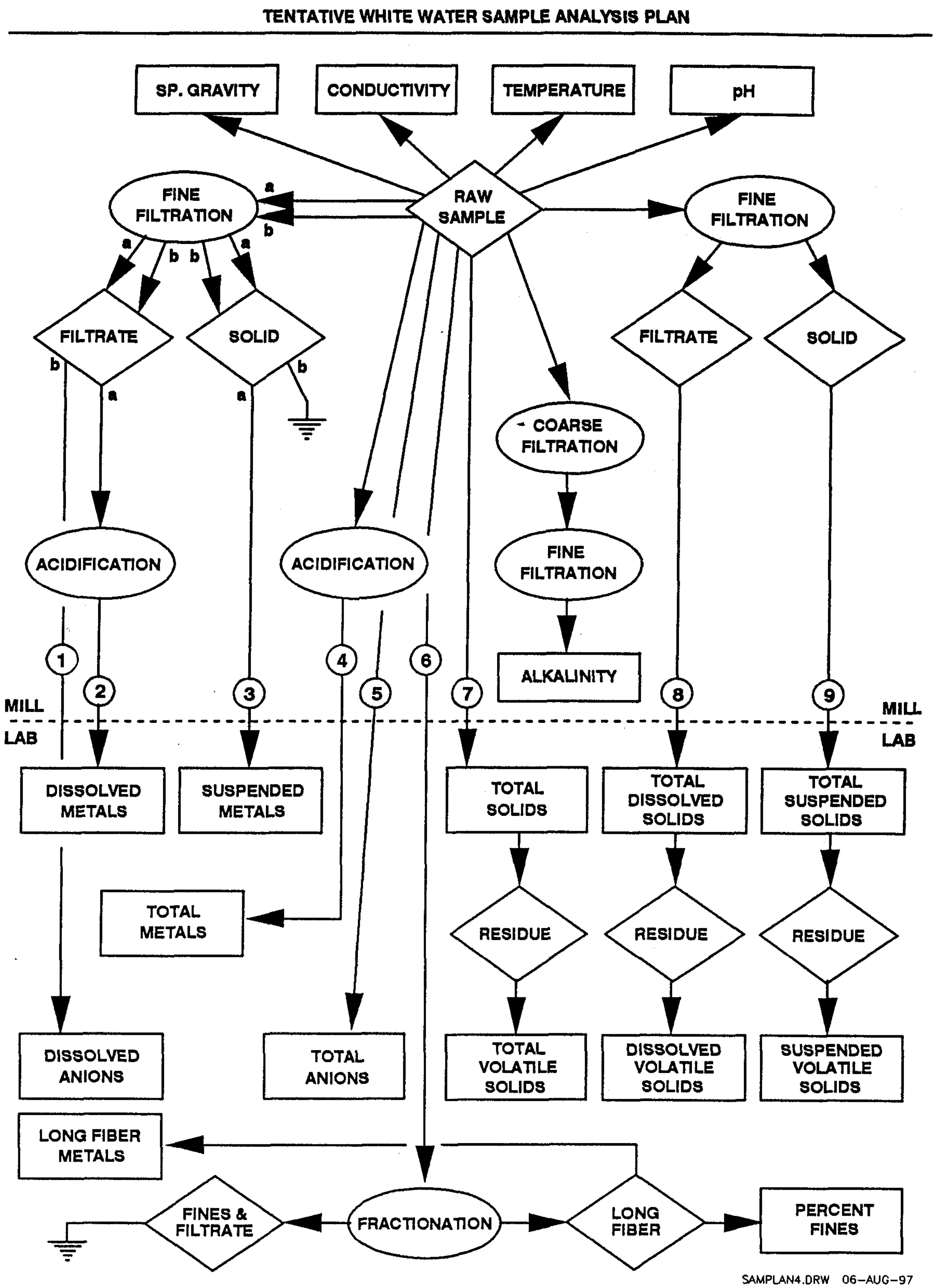

Figure 1. Schematic diagram of tentative white water sample handling and analysis plan.

internal consistency by measuring TS on an unfiltered raw sample. The residue left in the TS, TDS, and TSS determinations will be analyzed for volatile solids content to give a reasonable estimate of the organic-to-inorganic ratio in each sample fraction. The results of the field alkalinity test will be used in conjunction with pH, temperature, and TDS data to nomographically calculate carbonate, bicarbonate, and free carbon dioxide contents. The concentration of specific anions will be measured in both raw and filtered samples to determine if any anions are associated with the solids fraction.

Yet another aliquot of raw sample will be filtered in the field and the solid and acidified filtrate fractions will then be analyzed later in the laboratory for trace metal concentrations. An unfiltered raw sample will also be analyzed for trace metal concentrations as an internal consistency check. Finally, some acidified raw samples will be fractionated in a Britt Jar to determine percent fines. The long fiber fraction will be collected and analyzed for trace metals content, allowing us to calculate how NPE's partition between the long fiber-bound, the fines- and colloids-bound, and the dissolved phases. The extensive sample filtering outlined above and in **Fig. 1** will be conducted in the field to ensure that the measured phase partitioning of metal species will be representative of the actual white water composition at the time of sampling. The separation of suspended solids from dissolved solids will also reduce interference from possible metals migration between phases as samples cool, flocculate, sit in storage, etc.

Deliverable 3: Laboratory experiments to further define NPE behavior.

Laboratory experiments to evaluate metal binding to wood pulp were begun slightly ahead of schedule as part of the IPST *"Closed Mill"* matching project. Initial experiments were aimed at understanding calcium binding to wood pulp under controlled conditions. Data analysis based on the selectivity coefficient approach has been successful at modeling the calcium adsorption, suggesting that this approach may be potentially useful in equilibrium solver and mass balance calculations. Competitive experiments with sodium and magnesium have been completed and offer an explanation for the release of calcium at high pH.

An X-ray spectroscopy technique (i.e., Extended X-ray Absorption Fine Structure, EXAFS) has been applied to treated wood pulp and has proven to be a viable tool for characterizing the binding environment of tightly bound iron. This type of fundamental understanding of binding chemistry should prove to be useful in the mathematical modeling effort. Similar EXAFS experiments are planned in order to learn more about the environment of metal binding sites when they are occupied by manganese and calcium ions. A student has entered the IPST Ph.D. program with the intent of completing this X-ray effort and addititional funding from the Georgia Consortium has been secured to help defray the cost of using this analytical technique.

NPE Purge Evaluation Tasks

Many of the reported effects of paper machine white water system closure have been summarized briefly in **Attachment II.** It is obvious that some type of NPE purge technique will be necessary when NPE concentrations soar to problematic levels as mills move towards high degrees of closure. The laboratory work summarized below is based on the study of *model* white water. The determination of an appropriate chemical composition for the model white water was made after surveying the average white water compositional data present in the literature. This literature is shown tabulated in **Attachment II.** Also included in the attachment is a brief description of the primary purge technique of interest in this study: electrodialysis.

Deliverable 4: Laboratory experiments to determine precipitation behavior.

The influence of equilibration between model white water and atmospheric carbon dioxide on the precipitation of NPE's was investigated using laboratory experiments and solution equilibria simulation software (OLI, Inc.) Results indicated that the model white water was in equilibrium with atmospheric carbon dioxide during experimentation.

It is important that precipitation does not occur during fouling experiments to ensure that the fouling is being caused solely by the macromolecules as opposed to the precipitates. To this end, simulation software, provided by OLI Inc., and laboratory experiments are being used to verify that precipitation does not occur. The OLI software (ESP) successfully reproduced the solubility data of a mixed calcium chloride and sodium chloride salt solution found in literature and repeated in the lab. In addition, the OLI software successfully predicted the resultant pH, to two decimal places, of a laboratory solution composed of the model inorganics with a known amount of dissolved carbon dioxide.

Calibration procedures to detect one of the model foulants (i.e., sulfonated lignin) were established. A fluorescence technique was identified as the method of choice and excellent calibration was obtained.

Deliverable 5: Laboratory experiments to determine electrodialytic behavior.

Experiments on the fouling of ion exchanger surfaces with one of the model foulants (i.e, sulfonated lignin) were performed. The experiments were performed on beads made of the same material as ion exchange membranes. This was needed to obtain sufficient surface area for quantitatively measurable adsorption. Sulfonated lignin heavily fouls anion exchangers apparently irreversibly. The fouling of cationic exchangers is very low. Therefore, as expected, the anion exchange membranes will pose the greatest problem in the application of electrodialysis as an NPE purge for white water.

The membrane fouling characterization system (i.e., impedance testing of membranes) is nearing completion. The impedance testing system will allow us to distinguish between different organic and inorganic fouling mechanisms.

4. DELIVERABLES:

Major Milestones and Dates		Original Proposal	Actual
Computer simulation of wet-end(s).	start:	01-JUL-96	01-JAN-98
	complete:	31-MAR-97	30-JUN-98
Baseline sampling and testing.	start:	01-OCT-96	01-OCT-97
	complete:	31-DEC-97	31-MAR-99
Laboratory experiments	start:	01-APR-97	01-MAR-97
	complete:	30-SEP-98	31-DEC-98
Laboratory experiments to determine precipitation behavior.	start:	01-JUL-96	01-JUL-96
	complete:	31-DEC-97	31-DEC-97
Laboratory experiments to determine electrodialytic behavior.	start:	01-JAN-97	01-JAN-97
	complete:	30-JUN-98	30-JUN-98

5. BUDGET:

	Total FY 97	12-Month Expended
State Funds	$ 112,387	$ 31,246
Matching Funds	$ 55,645 *	$ 55,645

- The total amount of matching funds was listed as $64,000 in the original proposal (IPST Proposal No. 4475R1, 10-JUN-96). That amount, however, was incorrect because it was calculated based on an estimated overhead excess (i.e., above the 45% allowable) of 15%. The actual IPST "excess overhead" rate, as of 14-JUN-96, is 4.22%.

ATTACHMENT I

Time Table for Tasks Proposed for FY 1997 and Beyond

(Starting Date: 01-JUL-96)

	Year 1				Year 2				Year 3				Estimated FY97 Cost ($)
Project Quarter	1st	2nd	3rd	4th	1st	2nd	3rd	4th	1st	2nd	3rd	4th	
NPE Simulation Tasks													
Simulation of mill white water system	—	—	—										25,000
Baseline sampling, testing, and analysis		—	—	—	—	—							48,885
Laboratory experiments				—	—	—	—	—	—				30,000
Mathematical modelling								—	—	—	—		
Development of simulation module										—	—		
Model validation										—	—	—	
NPE Purge Evaluation Tasks													
Laboratory experiments in precipitation	—	—	—	—	—	—							} 8,500
Laboratory experiments in electrodialysis			—	—	—	—	—	—					
Development of precipitation purge					—	—	—	—	—	—			
Development of electrodialysis purge							—	—	—	—	—	—	

ATTACHMENT II

EFFECT OF WHITE WATER SYSTEM CLOSURE:

When white water is used where fresh water previously was added, three changes have been reported to occur to the white water. Increase in water temperature, total suspended solids (TSS), and total dissolved solids (TDS). Typical white water temperatures range from 110 to 120°F [1]. Temperatures have been reported as high as 160°F with closure efforts [2]. An increased white water temperature leads to increased drainage on the forming table. Increased temperature may also alter the population of biological growths. Efficient technology is already available for reusing suspended solids from the white water, so the TSS levels are not expected to increase significantly or otherwise be a problem. Going from a 0% closed system to a 100% closed system (reusing all water) could result in the TDS increasing by as much as 33-fold [3]. The TDS consists of organic macromolecules (organics) and inorganic salts (inorganics) (typically 500-5000 PPM TDS, 25-75% inorganic) [3-6].

Four problem areas are associated with increased levels of TDS: metallurgical, chemical, biological, and process/equipment related. Corrosion has been reported to increase with closure [3,6-8]. Chemical problems reported are precipitate/scale formation, decreased retention of paper fibers in the sheet that forms, decreased sizing (water resistance of the paper), loss of paper brightness, detrimental coloration, sheet mottling, and foam [3,6,8-13]. Increased levels of biological growths, slimes, and sheet odor have been reported [3,6,8,10]. Increased levels of dissolved solids can lead to increased felt plugging in the pressing operation to remove water from the paper [14,15]. For closure (reuse of white water rather than discharge) to proceed past the point where the above problems occur, a selective dissolved solids purge is needed. A separate purge for each of the organic and inorganic TDS fractions may be required. Closure to a specific discharge of 10 m^3/ton may be possible without encountering the problems above.

AVERAGE COMPOSITION OF DISSOLVED SOLIDS IN MILL WHITE WATER:

There is limited published information concerning white water organics. Dissolved organics can be introduced into the white water from the virgin pulping process, from the recycle pulping process, from chemical additives, and from the feed water. **Table 1** lists the average white water compositional data obtained from the literature [6,7,10,19-21,23-26]. The following abbreviations are used in the table: TS for total solids, DI for dissolved inorganics (ash), DO for dissolved organics, and Sp Disch for specific discharge. Also, 'Fine' stands for fine papers, 'Grnd/News' stands for ground wood and newsprint grades, and 'kft bd and pap' stands for kraft board and paper. The fine paper averages include data from mills with acidic white waters. Some average information concerning the inorganics is also listed in **Table 1**. Only sulfate, chloride, aluminum, and calcium concentrations were reported consistently. Below pH 7, carbonate species total less than 1 PPM in solution. Using simulation software (OLI Incorporated) and laboratory results, the aluminum was found to exist as an ionized species in solution at 23°C but > 99% precipitated as aluminum hydroxide at $\geq$ 25°C. Thus, all the aluminum in white water is expected to be present as a precipitate or otherwise not as an ionized species in solution.

Table 1. Average mill white water composition data.

Average concentration (ppm) breakdown by grade.

	Fine*	Grnd/News	krft bd & pap
TS	1800	5208	4114
TSS	1076	5084	2585
TDS	865	2268	1975
TDS/TS	0.48	0.44	0.48
DI	540	974	1361
DO	189	1054	428
DI/TDS	0.62	0.43	0.69
CO3		81	
SO4	345	540	790
Cl	28	22	24
Al	11	1	1
Ca	104	90	124
Na		250	
Sp Disch (m^3/t)	29	22	

Number of mills used for average.

	Fine*	Grnd/News	kft bd & pap
TS	22	10	102
TSS	24	8	90
TDS	23	16	104
TDS/TS			
DI	17	13	102
DO	17	12	102
DI/TDS			
CO3	0	2	0
SO4	21	9	102
Cl	24	9	102
Al	19	9	49
Ca	20	9	54
Na	0	2	0
Sp Disch (m^3/t)	2	3	0

* The Fine paper averages include data from mills with acidic white water systems.

MODEL WHITE WATER COMPOSITION:

The fouling studies are being conducted using a model white water. The model white water used for experimentation includes model organic and model inorganic dissolved fractions. The model organics are sulfonated lignin and anionic polyacrylamide. Sulfonated lignin was chosen to represent the worst case foulant (**Figure 1**). Sulfonated lignin possesses both aromatic and charged groups so that nonionic and ionic interactions with the aromatic and charged groups of the electrodialysis membrane are possible [22]. Arrows in **Figure 1** show the carbon atoms where sulfonation is expected to occur. It has been found that sulfonated aromatics are very serious foulants [17,18]. Anionic polyacrylamide is a high charge density polyelectrolyte that is used on paper machines. Polyacrylamide has no aromatic groups. Due to the higher charge density and no aromatic groups, it is expected that the polyacrylamide will exhibit a higher proportion of ionic adsorption than nonionic adsorption compared to the sulfonated lignin. The model white water inorganics are based on the averages found for unbleached kraft paper mills, which was the majority of literature data (**Table 1**). The average pH was 5.6, and the remainder of the positive charge is obtained using sodium. Inorganic precipitates are not desired during the experiments so the aluminum is not included in the model white water.

ELECTRODIALYSIS:

Current work is focusing on using electrodialysis (ED) as an inorganic purge (**Figure 2**). An ED stack is composed of compartments formed by alternating anion selective (A) and cation selective (C) membranes. A voltage gradient is applied across the compartments, which causes the charged species in solution to migrate toward the appropriate electrode. However, the arrangement of the membranes results in the ions concentrating in every other compartment while being diluted in the remainder. The attractive feature of ED is that the minority contaminant is removed from the bulk solution, compared to removal of the bulk water (membrane filtration, evaporation). Thus, minimal

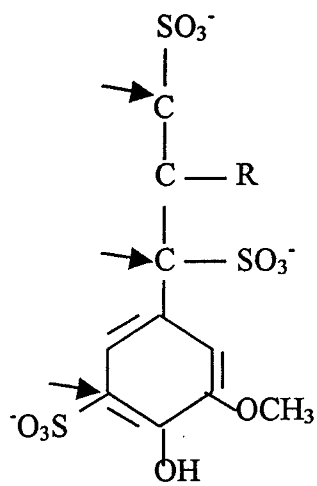

Figure 1. Sulfonated Lignin.

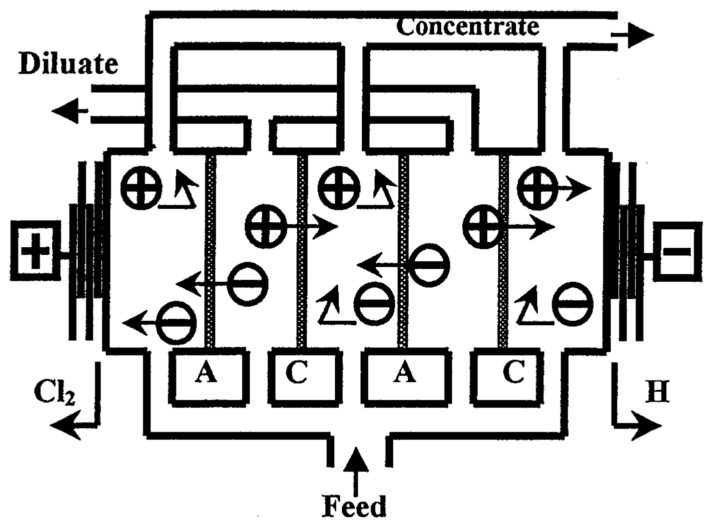

Figure 2. Typical Electrodialysis Stack.

energy is expended on the bulk stream. In addition, the ion exchange membrane is not regenerated as ion exchange resins. Fouling of the membranes, which is seen as a major concern for further applications of electrodialysis [16], is being investigated.

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