

ION-EXCHANGE TECHNOLOGIES FOR THE MINIMUM EFFLUENT KRAFT MILL

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ABSTRACT

Paper buying standards as well as environmental and economic factors are increasing the interest in progressing toward low effluent, closed-cycle kraft mills. For system closure to be realized, several problems need to be addressed. These include chemical imbalance (e.g. sodium/sulphur and caustic/chlorine) as well as build-up of non-process elements (e.g. chloride, potassium, calcium, magnesium, manganese, iron) in the recovery cycle. This paper will provide an overview of the technical feasibility, process integration and economics of three novel, cost-effective processes based on Eco-Tec's Recoflo ion-exchange technology, for addressing several of these problems.

A first process, called GAP (Generator Acid Purification), removes sulphuric acid from the sodium sulphate by-product of chlorine dioxide generators, thus avoiding the destruction of alkalinity in pulping liquors when this is added as sodium and sulphur make-up to the kraft recovery cycle. The purified sulphuric acid from this process can be returned to the chlorine dioxide generator or used in other mill applications. A second process, called PDP (precipitator dust purification), removes sodium chloride from the electrostatic precipitator dust of the recovery boiler thereby reducing the frequency of costly boiler shutdowns for washing off the low melting-point deposits in the flue gas passages. A third process, based upon sodium-cycle cation exchange, removes multivalent metal contamination from the acidic D-stage bleach filtrate, prior to recycle back to recovery, thereby minimizing scaling and other problems. This process which was recently installed at the Champion International, Canton, NC mill as part of their BFR (Bleach Filtrate Recycle) process, has seen almost continuous operation since the beginning of 1997.

INTRODUCTION

Accumulation of non-process elements (NPE's) is widely recognized as a major limitation to increased mill closure. Under some circumstances even process elements such as sulphur and sodium, which are necessary for pulping can build up to the point where the process is adversely affected. While a number of processes have been available for NPE removal for some time, less expensive and more efficient processes are needed to make closure more attractive and less problematic. It is even conceivable that some of these technologies could improve an existing operation, increased closure notwithstanding.

An alliance was formed in 1993 between the Pulp and Paper Research Institute of Canada (PAPRICAN) and Prosep Technologies Inc., a subsidiary of Eco-Tec Limited, to develop applications in the pulp and paper industry for Eco-Tec's novel ion exchange systems. These systems have been extensively used in a number of other industries since 1972 for recovery of waste chemicals and removal of non-process elements from process solutions. It was felt that this low-cost, highly efficient technology could be adapted to the pulp and paper industry through a joint research and development program.

RECOFLO ION EXCHANGE TECHNOLOGY

Most mill personnel are familiar with the large ion exchange demineralizers which are employed for purification of recovery boiler feed water. These systems typically utilize ion exchange columns 8-16 feet in height. The Eco-Tec systems utilize a patented¹ technology called *Recoflo*. Recoflo ion exchange demineralizer 'columns' to do the same job are only 6 inches in height as shown in Figure 1. In addition to the dramatic reduction in size, Recoflo demineralizers utilize only about one half the regenerant chemicals of a conventional co-current ion exchange system and produce higher purity water².

In addition to their short column height, Recoflo systems are characterized by a number of other features that differentiate them from other ion exchange systems. These include:

- counter-current regeneration
- fine particle size resins
- fully packed resin beds
- pre-assembled and pre-tested skid mounted construction
- very short cycle times in service and regeneration (i.e. minutes instead of hours)

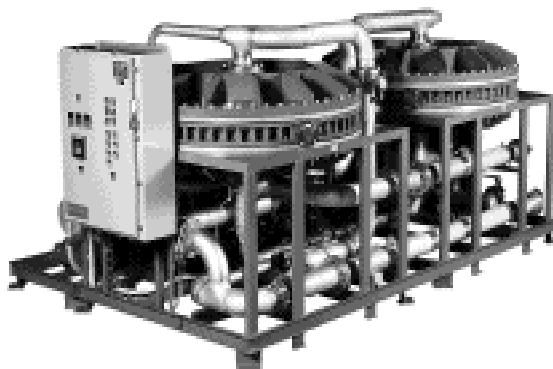


Figure 1: Typical Recoflo Ion Exchange Demineralizer

A large number of Recoflo demineralizers have been installed for production of high purity water for applications such as boiler makeup water in cogeneration plants. It is likely that the pulp and paper industry will see this technology utilized for recovery boiler makeup water production in the near future.

Although the benefits of small size and low chemical consumption for water demineralization are obvious, Recoflo technology was in fact originally commercialized for chemical recovery and purification because of its unique ability to treat and produce very concentrated solutions. This is in contrast to

conventional ion exchange technology, which is generally limited to processing dilute solutions. Some of such applications that have been extensively exploited include:

- recovery of metal salts such as chromium, copper, nickel, cobalt and zinc from electroplating rinsewaters
- removal of non-process elements from concentrated electroplating electrolytes
- purification of metal pickling, etching and anodizing baths
- recovery of sulphuric acid from metal refinery electrolyte bleeds

CHLORINE DIOXIDE GENERATOR ACID PURIFICATION

In 1977 Eco-Tec developed a system called the APU™ (Acid Purification Unit) for separation of metal salts from waste acid solutions. The APU utilizes an ion exchange resin which has the ability to sorb acids from solution, while excluding metal salt of those acids. This sorption is reversible, in that the acid can be readily de-sorbed from the resin with only water. It is thus possible, by alternately passing contaminated acid and water through a bed of this resin, to separate the free acid from the metal salt.

There are basically two steps in the APU operating cycle (see Figure 2): In the *upstroke*, acid containing dissolved metal salt impurities is pumped into the bottom of a fixed bed of sorption resin. The free acid is sorbed by the resin as the solution passes through the bed, so that a de-acidified metal salt solution is collected from the top of the bed. Next, during the *downstroke*, water is pumped down through the resin bed. Water strips the acid from the resin as it passes through the bed, so that purified acid is collected from the bottom of the bed. The total cycle time is typically only about 2-5 minutes. No chemicals or significant energy inputs are required to operate the APU process.

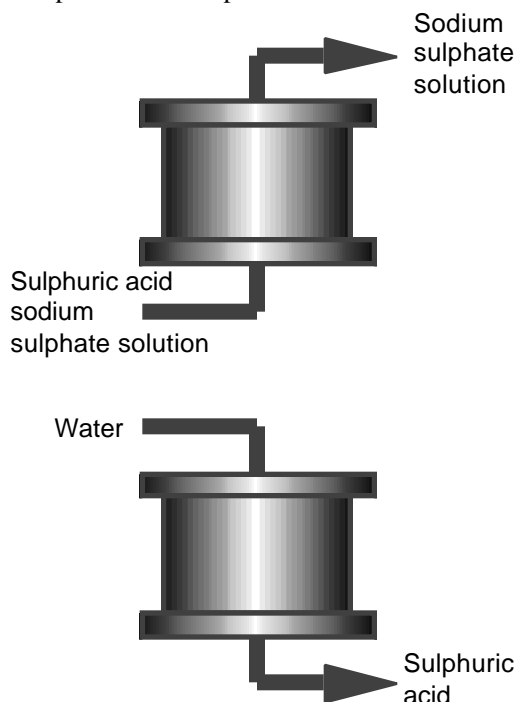


Figure 2: APU Operating Cycle

The APU was first commercialized for purification of sulphuric acid aluminum anodizing solutions³. Since that time, hundreds of units have been installed around the world on a wide variety of different acid recovery applications. A typical APU, which is shown in Figure 3 occupies less than 100 ft² of floor space, excluding auxiliary tanks.

The recent trend towards bleaching with chlorine dioxide has increased the quantity of byproduct acidic generator effluent produced, which must be disposed of. If all this material is recycled to recovery, one would expect to see a buildup in sulfur within the kraft cycle. Recently, the APU was adapted to treatment of acid effluents from chlorine dioxide generators. The system, known as **GAP (Generator Acid Purification)** separates free sulphuric acid from sodium sulphate⁴. The GAP system can be used with either atmospheric or vacuum generators.

With atmospheric generators such as Mathieson or Solvay, the generator effluent is a concentrated liquor containing sulphuric acid and sodium sulphate with a low residual concentration of sodium chlorate. To avoid oxidation of the APU resin, the residual chlorate in the generator effluent is first reduced with a chemical reducing agent such as sulfur dioxide⁴. The treated solution is then fed through a filter to remove any suspended solids and on to the APU. The free sulphuric acid is taken up by the resin and a de-acidified sodium sulphate is collected. This sodium sulphate is recycled back to the recovery cycle. Since most of the sulphuric acid has been removed, the amount of caustic soda necessary to neutralize the acid that accompanies the sodium sulphate is substantially reduced. These savings go a long way towards paying off the capital investment in the GAP system.

Purified sulphuric acid is recovered by water elution of the APU resin bed. This recovered sulphuric acid can be reused in various ways. The obvious approach is to recycle the acid back to the chlorine dioxide generator. In this case a small vacuum evaporator is installed to concentrate the reclaimed acid back up to about 60% w/w. Many mills may be able to use the dilute acid for applications such as brown stock washing or tall oil acidulation so that an evaporator is not required.



Figure 3: Typical APU Used for Generator Acid Purification

Typical APU results from a GAP application are shown in Table I. The GAP system gives an atmospheric generator the operating cost benefits of a vacuum unit (e.g., SVP-Lite, R-8) without the high cost of equipment replacement. In fact, a GAP system can yield the same acid recovery efficiency as vacuum generators with salt cake washing equipment (e.g. SVP-SCW, R-10).

Table I: Typical GAP System Performance
basis: 20 TPD ClO₂ Production (Mathieson Generator)

| | Na ₂ SO ₄ (g/L) | H ₂ SO ₄ (g/L) | Flow (L/h) |
|--------------------|--|---|---------------|
| generator effluent | 288.0 | 453.5 | 4560 |
| purified acid | 23.8 | 302.6 | 6125.8 |
| de-acidified salt | 128.1 | 27.5 | 9188.8 |
| evaporator product | 70.7 | 900.0 | 2059.6 |

The GAP system can also be used with sub-atmospheric generators such as Eka Chemical's SVP-Lite™ and Sterling Pulp Chemicals R-8. The system is then called GAP-S, and is an attractive alternative to a salt cake washer or R-10 upgrade. The flowsheet for the GAP-S system is shown in Figure 4.

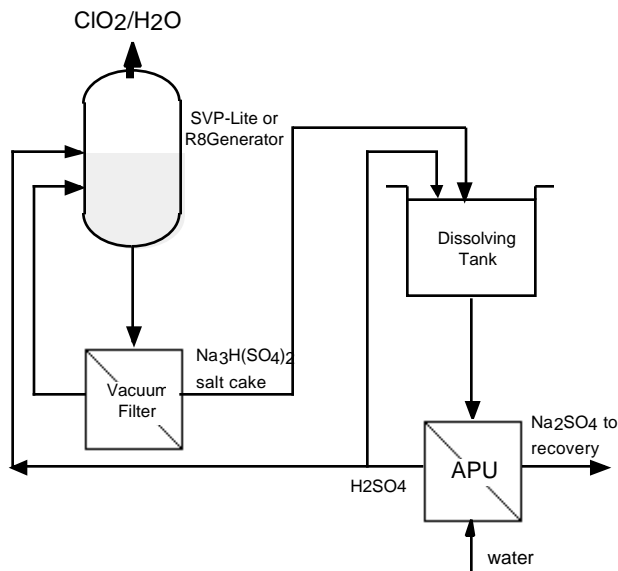


Figure 4: GAP-S Acid Purification System For Vacuum ClO₂ Generators

In the GAP-S system the sodium sesquisulphate salt cake obtained from the vacuum filter is first fully dissolved. The solution which contains sulphuric acid and sodium sulphate is then fed to an APU. Due to the lower sulphuric acid concentration of the solution, destruction of the small amount of residual chlorate present is not necessary. As with the basic GAP system, the deacidified sodium sulphate byproduct is recycled back to recovery. A portion of the purified dilute sulphuric acid obtained by water elution of the APU resin is recycled back to the dissolving tank to dissolve the sodium sesquisulphate. By this means the acid concentration in the solution is fortified beyond what is possible by simply dissolving the sesquisulphate in water. The balance of the sulphuric acid eluate is recovered and

recycled back to the generator. Vacuum generators will often have sufficient evaporative capacity to handle the additional water load. If not, it may be possible to upgrade the evaporation capacity of the generator at nominal cost. Because an evaporator is generally not required, capital costs for GAP-S are significantly less than for a GAP system.

Typical performance for a GAP-S system is shown in Table II. While the GAP-S system provides similar recovery performance levels to a salt cake washing system, it is much more compact and less complex to operate.

Table II - Typical GAP-S Performance
basis: 20 TPD ClO₂ Production (SVP-Lite or R-8)

| | Na ₂ SO ₄ (g/L) | H ₂ SO ₄ (g/L) | Flow (L/h) |
|-------------------|--|---|------------|
| purified acid | 81 | 126 | 1715 |
| de-acidified salt | 308 | 7 | 2899 |

CHLORIDE REMOVAL FROM ESP CATCH

High levels of chloride and potassium impurities in kraft liquors are known to accelerate recovery boiler plugging under some circumstances. The current levels in some mills already require that boilers be shut down for water washing as frequently as every three months. As mills move towards higher levels of closure the current outlets for these elements are reduced. This will result in even higher chloride and potassium concentrations throughout the kraft cycle. Therefore, to reduce lost production due to boiler shutdowns control of these elements will be necessary in many cases. In fact, elimination of only one boiler shutdown per year for clean-out could easily warrant installation of a chloride removal system.

The sticky temperature of the flue dust in a recovery boiler is defined as that temperature where the amount of liquid in the material is high enough for the mixture to stick on a metal surface. Laboratory and mill measurements have shown that the sticky temperature is reached when approximately 15% of the mixture becomes molten. Rapid plugging of the recovery boiler can occur if the flue gas temperature exceeds T₁₅ in the generator bank.

As Figure 5 shows, the chloride and potassium levels of the recovery boiler tube deposits have a significant effect on the sticky temperature, T₁₅. If the chloride level can be reduced below 5% mole fraction, the sticky temperature should significantly increase and a reduced tendency for boiler plugging may be experienced. For example, at 5% K, by reducing the chloride level from 5% to 1.5%, the sticky temperature would increase from about 620°C to 740°C. It is important to note that when the deposit chloride levels are low, (i.e. less than 1.6 mole%) potassium has very little effect on the sticky temperature.

These conclusions have been confirmed recently by other workers^{6,7} and by mill practical experience. Many mills experiencing recovery boiler plugging problems have elected to discharge a portion (typically 15-50%) of their ESP catch, to provide an additional purge of chloride from the recovery cycle. These mills were able to achieve longer operation time between boiler water washes and chill-and-blow cleanings within a couple of months. They were also able to identify economic benefits resulting from the reduced chloride level in

the black liquor: eg. higher boiler efficiency, increased boiler throughput, higher steam temperature, lower sootblower maintenance and steam consumption. Of course, there is a penalty to pay for these benefits in terms of increased chemical consumption (mainly caustic soda).

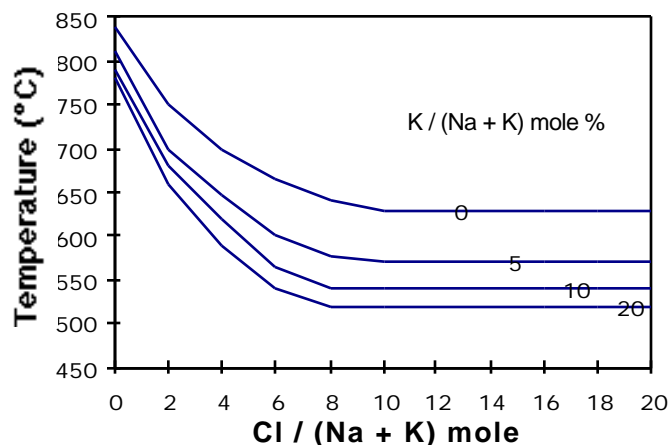


Figure 5: Effect of Chloride and Potassium on Deposit Sticky Temperature⁵

The Recoflo technology has recently been adapted into a new unit called an *SSU* (*Salt Separation Unit*) for separating different types of salts. The basic SSU process and equipment is the same as the APU shown in Figures 2 and 3, however the SSU employs a new type of sorption resin. The SSU resin is a special ion exchange resin with both cation and anion exchange groups existing on each resin particle. This resin can therefore remove both anions and cations *simultaneously*.

The SSU resin has unusual selectivities compared to conventional ion exchange resins. For example, the SSU resin has a very high selectivity for sodium chloride compared to other chemicals such as sodium sulphate and sodium carbonate. As a result, the SSU can remove sodium chloride from even concentrated solutions of sodium sulphate. The resin is very stable and under normal conditions should have an operating life time of many years. APU resins, which are very similar in composition and operated under similar hydrodynamic conditions, have shown lifetimes of 5-10 years in much harsher environments. As with the APU, the SSU resin is regenerated with only water. No chemicals or significant energy inputs are necessary. The process is therefore very inexpensive to operate.

It is well established that chloride becomes enriched in the electrostatic precipitator dust. This is therefore the logical place from which to attempt removal of chloride. A precipitator dust purification (PDP) system utilizing the SSU is shown in Figure 6. The dust is first dissolved in warm (i.e., 40-60°C) water to produce a 28% sodium sulphate/sodium carbonate solution containing chloride contamination which is just below the saturation limit. The precipitator dust solution is fed to a pressure 'pulse' filter where insoluble solids, such as metal oxides and any carbon material, are removed. This filter is similar to the white liquor pressure filter frequently used in many kraft mills. Filtrate overflows from the filter's collection chamber and is directed to the feed tank for treatment by the SSU. A typical back-pulse cycle for the pulse filter would be every 10 minutes and would

last about 15 seconds. During back-pulse, filtrate flows in reverse direction through the filter thereby removing solids from the filter elements. The solids then settle to the bottom of the filter vessel where they thicken to a concentration of 10-20%. Periodically, bottoms from the filter are withdrawn and either purged from the system or bypassed around the SSU to the sulphate product tank, before recycle back to the recovery cycle.

As with the APU (acid purification unit) described above, there are two basic steps in the SSU operating cycle: upstroke and downstroke. During the upstroke, filtered ESP dust solution from the feed tank is pumped up through the SSU resin bed. Sodium chloride is taken up by the resin and a purified sodium sulphate/carbonate solution is withdrawn from the top of the bed and collected in the sulphate product tank. In the downstroke, warm water is pumped into the top of the bed eluting sodium chloride from the resin. A portion of the chloride waste collected from the bottom of the bed is recycled back to the dissolving tank to dissolve more dust and the remainder is purged from the system.

Typical performance of a PDP system, based upon laboratory pilot plant runs, is shown in Table III. Dust samples from a number of different mills have been processed with comparable results. Note that the system removes as much as 97% of the chloride while recovering up to 99% of the sulphate and carbonate values. This compares very favorably with results recently reported⁸ on evaporative crystallizer systems which show only 80% sulphate recovery and 90% chloride removal. The SSU does not show any selectivity for potassium, although as discussed above, this should not prove to be an issue for most mills. Potassium removal for an inland mill would typically be about 5 - 10% while for coastal mills with higher chloride levels, the potassium removal would increase to about 10 - 15%.

Table III - Precipitator Dust Purification System Results
basis: 100 TPD ESP catch (typical inland mill)

| | |
|-----------------|-------|
| Na | 33.6% |
| K | 7.6% |
| SO ₄ | 49.4% |
| CO ₃ | 7.0 |
| Cl | 2.5% |

| | Flow (L/h) | [Na] (g/L) | [K] (g/L) | [SO ₄] (g/L) | [CO ₃] (g/L) | [Cl] (g/L) |
|---------|------------|------------|-----------|--------------------------|--------------------------|------------|
| Water | 15,948 | | | | | |
| Product | 10,750 | 111.4 | 24.6 | 172.0 | 24.4 | 0.3 |
| Waste | 6,557 | 11.0 | 2.1 | 2.7 | 0.2 | 14.0 |
| %Rem. | | 5.7% | 5% | 1% | 0.5% | 97% |

While the SSU itself does not consume any significant amounts of energy for its operation, recycle of the purified salt cake solution will impose an additional evaporation load on the black liquor evaporators. To determine the impact of this load a typical mill is assumed to process 1,750 kg of black liquor solids (BLS) per tonne of pulp. Approximately 5% - 6% of this material, 96 kg-BLS/tonne-pulp, is captured as ESP dust and processed by the PDP system. The purified salt cake solution recycled for recovery is 28% w/w and therefore the total additional evaporation required is roughly 235 kg-water per tonne of pulp if 100% of the dust is processed. In many cases processing

only a portion of the dust collected will suffice and the water load will be reduced proportionately. To avoid any precipitation or scaling, the point at which this solution is introduced must be carefully selected.

A computer based model has recently been used to predict the impact of the PDP process on an actual kraft mill⁸. Data collected from Georgia-Pacific's Leaf River mill in New Augusta, MS were used as the basis for this model. This ECF mill processes softwood and mixed hardwood to produce 1,630 ADMT/d of pulp. A best management practices (BMP) case was considered where the screen room has been closed and liquor losses reduced to give a soda make-up of 8.66 kg-Na₂O/ADMT. The results of this simulation for treatment of all the ESP dust are shown in Table IV. It should be noted that the mill data used for the simulation were collected from a softwood run.

As expected, the simulation predicts a large reduction in the chloride content of the ESP dust and black liquor, but a much smaller drop in the potassium when treated by the PDP process. While treatment by evaporative crystallization removes 90% of the potassium, a comparison of the sticky temperatures for these processes shows there is no significant difference. This again illustrates the minor influence of potassium on sticky temperature at low chloride levels.

Table IV - Simulation of PDP Impact on Kraft Process⁸

| | BMP Base Case | With PDP | With Evaporative Crystallizer |
|---------------------------------|------------------|----------|-------------------------------------|
| Cl in fired black liquor (%w/w) | 0.76 | 0.084 | 0.087 |
| Cl/(Na+K) in ESP dust (mole%) | 5.8 | 0.66 | 0.75 |
| K/(Na+K) in ESP dust (mole%) | 11.8 | 9.1 | 2.1 |
| ESP dust sticky temp. (C°) | 565 | 770 | 790 |

Even though simulation data and literature references document the greater impact of chloride on plugging, some mills, those processing hardwood for example, may require more potassium removal than is currently possible using the PDP system. If this is the case, it should be noted that up to 20% of the dust could be bled from the system while still giving the same sulphate recovery efficiency as the evaporative crystallization process. This would then allow more than four times the current potassium removal efficiency. Work is also currently underway to develop a potassium removal system that could be added on to the PDP system for those mills that do require more K removal.

A separate project is also underway to evaluate the use of the SSU for chloride removal from make-up caustic. This would of course reduce the overall input of chloride to the kraft mill and offers another approach to chloride control.

The PDP system has a number of significant advantages compared to other competitive technologies currently proposed for precipitator dust purification, such as

evaporative crystallization and electro dialysis. These include:

- significantly lower capital and installed cost
- lower operating cost
- better sodium sulphate and carbonate recovery efficiency
- better chloride removal efficiency
- significantly less floor and head space

METALS REMOVAL FROM BLEACH FILTRATES

Calcium, manganese and other multi-valent mineral non-process elements (NPE's) enter the mill via wood, water and chemicals. Normally, these impurities leave the mill in grits and dregs as part of the causticizing operation, as well as through inadvertent losses of cooking liquor. Loss of impurities to the bleach plant via the brownstock also provides a significant purge in normal operations. During contact in the acidic bleaching stages, metals are leached from the pulp fibres into the bleach filtrates. When bleach filtrate streams are recycled, NPE's accumulate. As levels increase, mineral deposits (scale) form in pipes and equipment.

Ion exchange-type water softening systems have long been used to prevent mineral scaling in low pressure boilers. The process is similar to the cation exchange portion of a water demineralizer except that the ion exchange resin is regenerated with sodium chloride instead of acid. The scale causing metals such as calcium and magnesium are then exchanged with sodium on the resin. The advantages that Recoflo offers the demineralization process (i.e.. compact size, low operating cost) are also available for water softening.

Removal of multi-valent metal NPE's to avoid scaling was recognized by Champion International to be an essential requirement for its BFR® bleach filtrate recycle system. After evaluating various alternatives, Champion elected to install an Eco-Tec Recoflo cation exchange system at its Canton NC BFR demonstration plant. In this installation, which employs oxygen delignification and an ECF bleaching sequence, a portion of the acidic D1 filtrate is passed through the metal removal process (MRP) as shown in Figure 7. The balance of the D1 filtrate is used on the fiber line pre-washer.

The MRP consists of a coarse strainer to remove large fibers, followed by three multi-media filters (two operating in parallel, one in backwash) for removal of the remaining suspended solids. The filtered solution is then passed through two parallel cation exchange units (IX2, IX2), each employing resin beds 60 inches in diameter by 6 inches in height. One of these units is shown in Figure 8. The total net flow that can be treated by the MRP is approximately 800 gallons per minute when fully operational. The purified D1 filtrate is recycled to the D1 washer lower pond.

The multi-media filters are backwashed several times each day with mill water to waste. Typical of Recoflo, the ion exchange units have very short cycle times. The units are normally in service for only 5-6 minutes and offstream, for regeneration, for 1-2 minutes.

The MRP was commissioned in November 1995. Although a number of mechanical problems were experienced during the initial operation, these have been worked out and the system is currently working reliably. Filtration, in particular proved to be a significant challenge due the unexpectedly high concentration of fine fibrous contamination

in the filtrates. Champion has reported^{9,10} that the system has met the original objectives by removing 85-90% of the hardness (i.e. calcium, manganese, magnesium) from the D1 stage filtrates over extended periods of operation during 1997.



Figure 8: Recoflo Ion Exchange Unit Installed at Champion, Canton NC

SUMMARY AND CONCLUSIONS

New ion exchange systems have been developed which are capable of removing impurities from pulping and bleaching liquors and adjusting chemical balances. These systems are unique in that they all utilize extremely small ion exchange columns and employ little or no chemicals for their operation. The small space requirements and low operating costs that result should make them very attractive to the industry. While these systems address a number of key concerns relating to increased mill closure, in order to successfully integrate them into a mill it is essential to consider what secondary effect operation of these systems may have.

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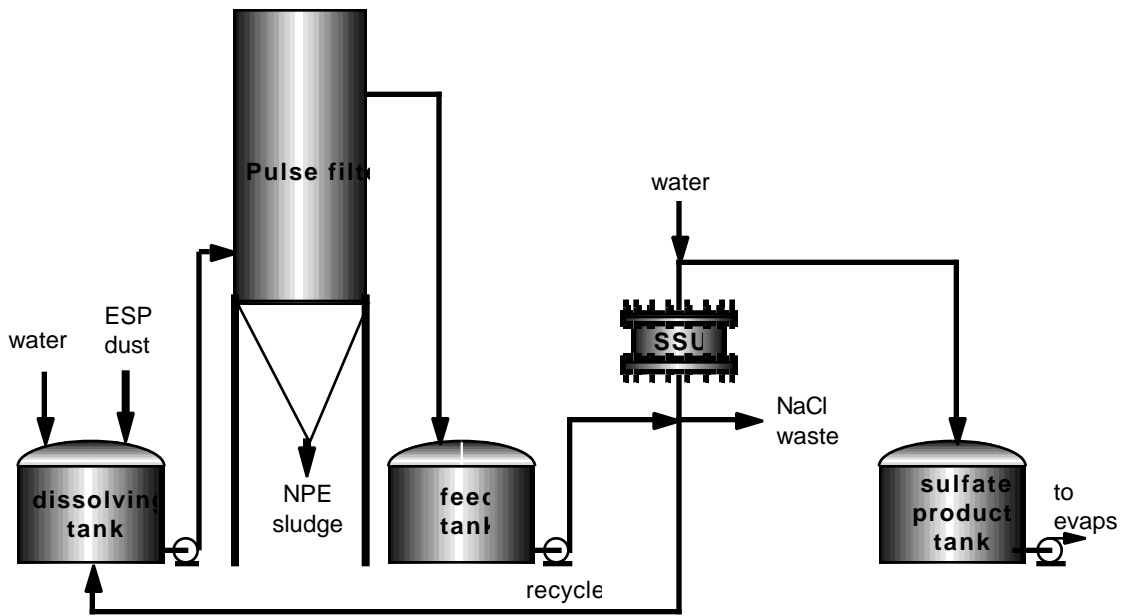


Figure 6: Precipitator Dust Purification System

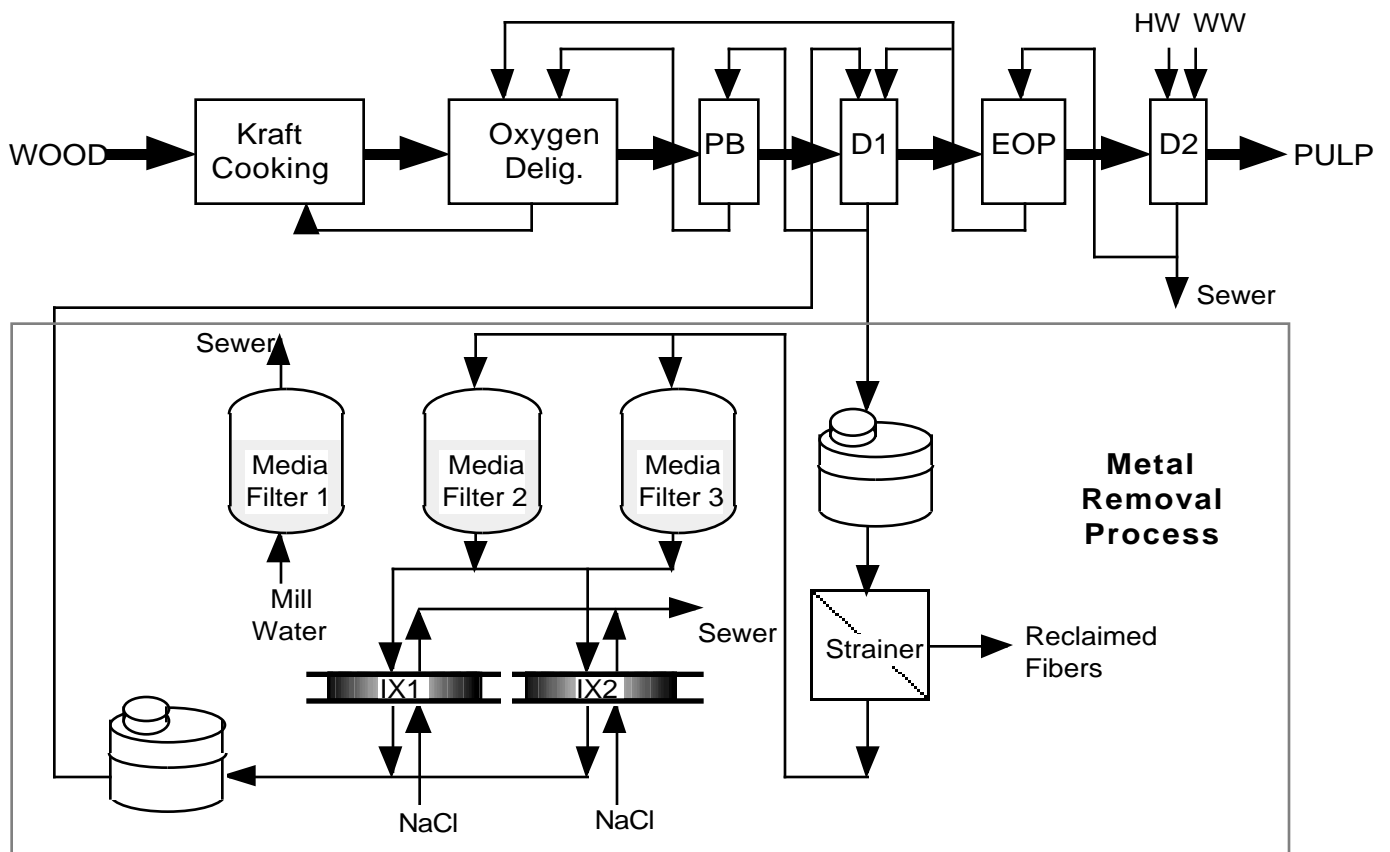


Figure 7: Metals Removal Process at Champion