BENCHMARKING AND MODELING OF A CLOSED-LOOP PILOT SYSTEM

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Abstract
Computer modeling and tracer studies are useful tools for benchmarking and characterizing closed-loop paper recycling systems. Mills can benefit from these tools by using them to investigate and solve problems related to contaminant removal/cycle-up of contaminants, yield loss, brightness reversion, and holding times. Forest Products Laboratory (FPL) is using these tools specifically to examine contaminant cycle-up time and arrival to steady state concentration in their closed-loop recycling pilot plant.

Closed-loop recycling system has been modeled using a general mass balance modeling program. Following model development, pilot plant trials were conducted using a tracer species to test the model. The goal of these trials is to monitor the tracer concentration in clarified process water and compare trial results with model predictions. This paper will introduce the methods employed, present data and results, and provide specific examples of how mills can use these tools for their benefit.

Introduction
Our pilot plant is being used to study the behavior of species during recycling in a system with a closed-water loop. There is interest in how the contaminants in process water affect the behavior of pressure sensitive adhesives (PSAs). An important consideration for these studies is the time to steady state concentration for the various species in the system. Possible species of interest are:

1. Pulp fines
2. Inorganic particles (clay, calcium carbonate, etc.)
3. Polymeric particles (adhesives, binders, etc.)
4. Colloidal polymeric particles (sizing, tackifiers, surfactants, etc.)
5. Dissolved polymeric materials (starch, modifiers, etc.)
6. Dissolved inorganic materials (salts, counter-ions, etc.)

The time to steady state depends on the concentration at start-up, flow rates, tank volumes, split ratios, and removal efficiencies of each unit operation. Since these are not the same for all species, there is not a single time to steady state but a time associated for each species. Furthermore, the concentration of a particular species is not uniform throughout the system, so the time to steady state also depends on where in the system it is measured.

Before testing adhesive behavior, the pilot plant system was fully characterized using modeling and tracer trials. This paper describes the comparison of the model predictions to trial results.

Closed-Loop Process
The paper recycling pilot plant has seven stages: (1) high consistency pulping; (2) pressure screening; (3) forward cleaning; (4) through-flow cleaning; (5) flotation; (6) washing/dewatering; and (7) water clarification. A diagram is shown in Figure 1. The process is started with fresh soft water, but once generated, clarified process water is recycled for use in pulping of subsequent batches and as dilution water for pressure screening and cleaning stages. Plant operations are typically performed at 46 ± 2°C. Process temperature is maintained by temperature controllers and heat exchangers. A data acquisition system records flow rate at 33 locations in the process, temperature at four locations, pulper power, and clarifier inlet and outlet turbidities.

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High Consistency Pulping
Each batch of pulp consists of 150 kg OD material composed of 5% pressure sensitive adhesive (PSA) coated material with the balance being a 1:1 mix of virgin copy and envelope papers. This total mix gives a mass loading of 1% adhesive. Each batch is pulped to 14% csc. for 20 minutes in a Voith HC 1.5 pulper. This pulp is then diluted to 2.5% csc. Pulper batches are timed to allow continuous operation of subsequent unit operations. Fresh water is used in the first batch and clarified process water is used for all subsequent batches.

Pressure Screening
The pressure screening stage consists of four slotted pressure screens in a feed-back, feed-forward configuration. Feed to the first primary pressure screen (0.3 mm milled slots) is diluted in-line to approximately 1% csc. Rejects are fed to a secondary screen (0.2 mm c-bar slots). Accepts from the first primary and two secondary screens are collected, diluted to 1% csc. and fed to the second primary screen (0.15 mm c-bar slots), with the final accepts being fed to the cleaning stage. The rejects from the second primary screen are fed to another secondary screen (0.15 mm c-bar slots). Screen banding is used to increase passing speeds to 1 m/s.

Forward Cleaning
The forward cleaning stage consists of two primary forward cleaners (Celleco 270) and one secondary forward cleaner (Voith).

Through-flow Cleaning
The through-flow cleaning stage consists of two stages of through-flow cleaners (first stage: Black-Clawson Xclones; second stage: Beloit Uniflows).

Flotation
The flotation stage consists of a 2,000 L two-stage La Mort vertical flotation cell. The flotation aid is DI-700, a nonionic surfactant that is added at 0.1% loading. Flotation rejects are sent to the drain and accepts are sent to the washing/dewatering stage.

Washing
A pilot drum washer having a 75-mesh screen is used to dewater the pulp to approximately 24% csc.

Clarification
Drum washer effluent and through-flow cleaner rejects are sent to the dissolved air flotation unit (Poseidon PPM-25) for process water clarification and recovery. A coagulant (Cytec C-577) and a flocculent (Cytec Superfloc 4818) are added to the feed in concentrations of 6 ppm and 10 ppm, respectively. The clarified process water typically has a turbidity of 35 NTU or lower and a flow rate averaging 380 Lpm. Clarified process water is collected in a holding tank equipped with a heat exchanger to maintain temperature near 46°C.

Computer Model
WinGEMS (Pacific Simulations Inc.) is a general energy and mass balance modeling program. Known parameters such as flows, consistencies, tank volumes, efficiencies, and temperatures for the various unit operations are specified in a basic block diagram that includes each unit operation. Figure 1 shows the block diagram that represents the closed-loop process. For the purposes of this model, volumes of nine tanks, flotation cell, and clarifier were considered as listed in Table 1. The volumes of the pipes were added to associated tanks. All of the tanks were considered to be well stirred. Both the flotation cell and clarifier were modeled as a combination of plug flow and well-stirred tanks.

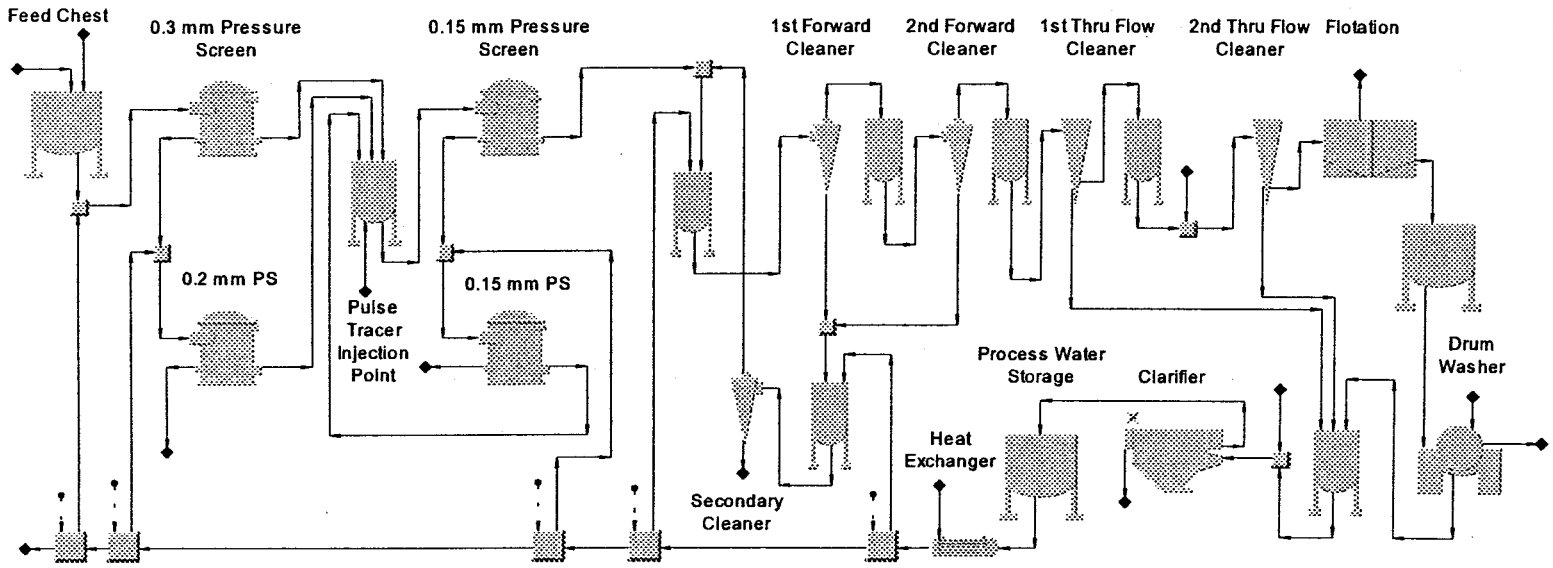

Figure 1: Flowsheet used for simulation studies.

Table 1 : Volumes used for the simulation

Tank Identification	Vol. (L)
Screen Tank	120
1 st Forward Cleaner Feed	300
Secondary Cleaner Feed	70
2nd Forward Cleaner Feed	500
1 st Through-Flow Cleaner Feed	310
2nd Through-Flow Cleaner Feed	400
Flotation Plug	500
Flotation CSTR	1500
Washer Feed	1700
Washer Seal	380
Clarifier Plug	470
Clarifier CSTR	5600
Water Tank	3000

Once the computer model of the system is generated, it can be used to predict the movement of chemical species though the pilot system. As a test of the model, two types of tracer experiments were conducted: one where a pulse of tracer was added to the dilution tank in the screening stage and the other where tracer was added to each pulper batch.

Tracer Studies

In both tracer experiments potassium was selected as the tracer species since it is not selectively removed by any unit operation in the closed-loop recycling system, and thus, gives a worst-case time to steady state.

Pulse Experimental Method

For the pulse tracer trials the closed-loop system was started and run with just water (no pulp) but with flow rates matching that of a typical closed-loop trial. At the five-minute time point two kg of potassium chloride (KCl) dissolved in 20 L of water was added to the tank feeding the primary 0.15 mm (6 cut) screen, indicated by the label "Pulse Tracer Injection Point" in Figure 1. Water was drawn from the following sample points: (1) 2nd forward cleaner accepts; (2) 2nd through-flow cleaner accepts; (3) flotation accepts; and (4) clarifier accepts. The time points for each sample point were chosen to best catch the spike in potassium concentration as it moved through the system. Approximately 72 water samples were taken and submitted for analysis by inductively coupled plasma emission spectroscopy (ICP). In the concentration range of these samples, ICP gives a coefficient of variation of approximately 1%.

Pulse Experimental Results

Four pulse trials with potassium were conducted. Three were considered to be of sufficient quality for further analysis. Figure 2 shows the results of one trial.

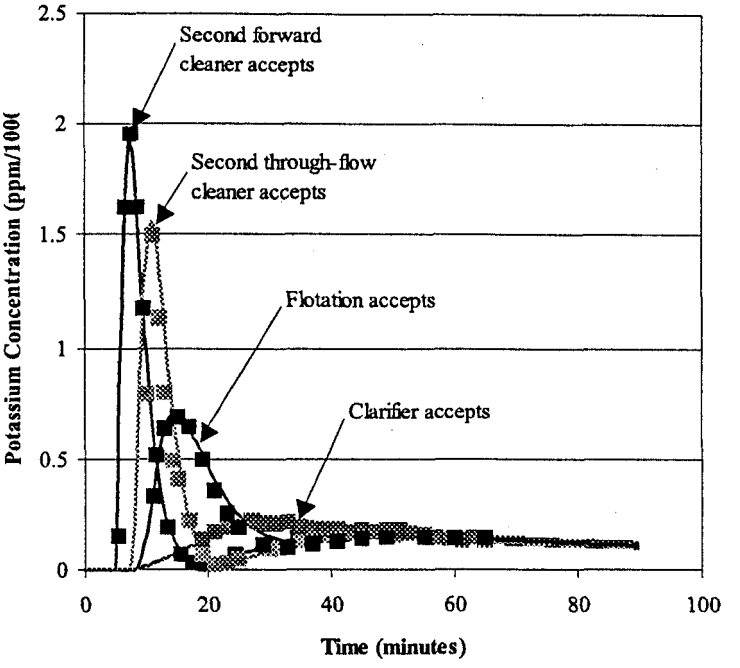

Figure 2: Potassium concentration as a function of time. The data are represented by symbols and the lines are the model.

Inspection of Figure 2 indicates that the model can accurately predict both the height and the position of the peaks in potassium concentration. Furthermore, the second peak caused by potassium coming back from the clarifier in the recycled clarified water is also accurately modeled.

Steady-State Tracer Studies

The same model used for the pulse experiments was used to predict arrival to steady state after a step change in the concentration of a component entering with the pulp via the feed chest in Figure 1. Figure 3 shows the results of 10 simulations of the concentration of a species in the clarifier accepts. As expected, inspection of Figure 3 shows that as removal efficiency is increased time to steady state is decreased.

These model data indicate the relationship between removal efficiency and time of trial required for the closed-loop system. Removal efficiency in this model represents overall efficiency in the system. The model suggests that there is not a significant difference between removal by flotation and removal by clarification. The model predicts that if all the species are removed at a 90% level by any unit operation in the system, then two batches of pulp (or about two hours) should be sufficient to achieve steady state. If, however, any important species is removed at a lower efficiency then the trial time must be extended.

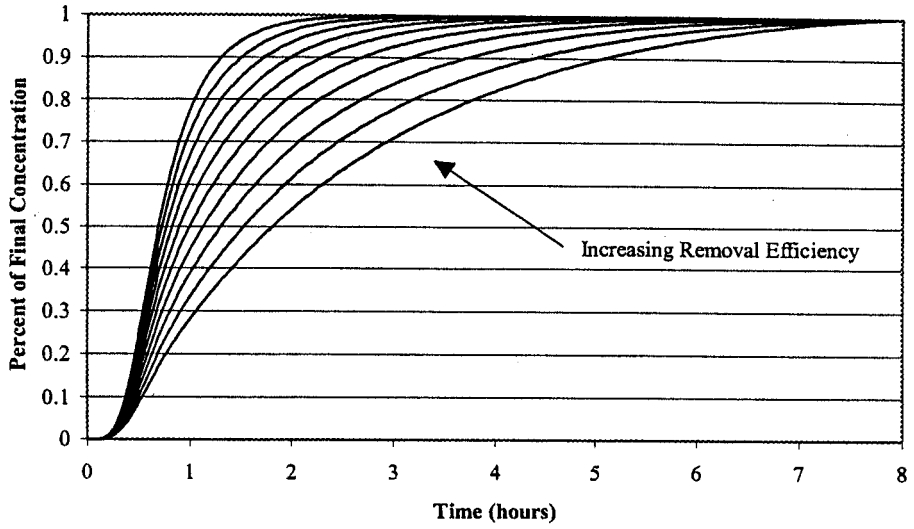

Figure 3: Model predictions for relative concentration of the clarifier accepts for a species that has various system removal efficiencies (0% to 90%).

Steady-State Tracer Trial Experimental Method

As with the pulse experiments, potassium was used as the tracer species. Since it is not selectively removed by any unit operation, it will take the longest time to reach a steady-state concentration. There is a small effect due to potassium adsorption on pulp, but this effect is not considered for this study and potassium is assumed to split with the water phase in all unit operations.

For these trials two kg of potassium chloride (KCl) diluted in 10 L of water was added to each pulper batch. Samples of the clarifier effluent were taken every five minutes. After filtering through a 0.45-micron filter, the samples were analyzed for potassium by inductively coupled plasma emission spectroscopy (ICP). Chemical oxygen demand (COD) was also determined for the filtered samples using Standard Methods 5220 D Closed Reflux, Colorimetric Method.

Steady State Tracer Trial Results

Three steady state tracer trials were conducted, two that included two pulper batches and one that included four pulper batches. Figure 4 shows the potassium concentration of the clarified effluent over time for the trial that included four pulper batches with post-consumer benchmark adhesive. The total duration of the trial was 270 minutes. The benchmark adhesive is an acrylic material that has adequate screenability. The term “post-consumer” refers to adhesive material affixed to envelope paper without the silicone release liner present.

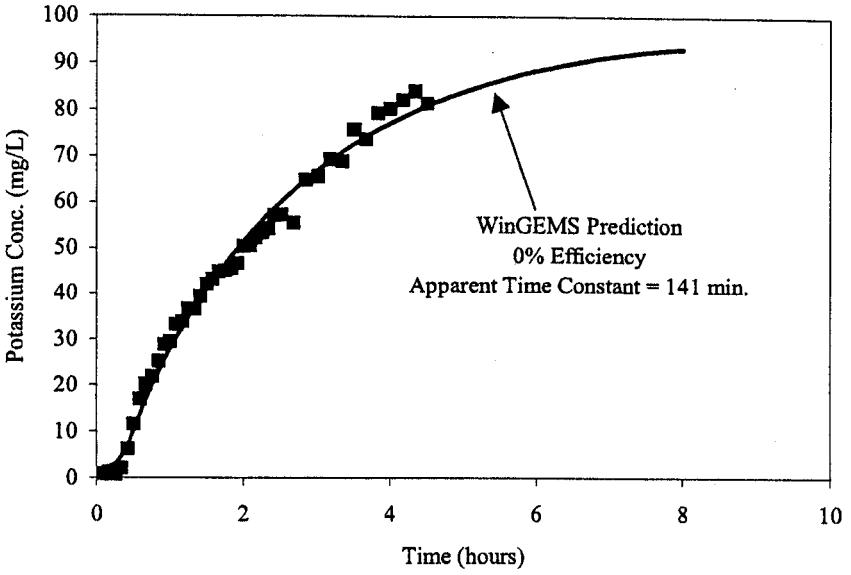

Figure 4. Potassium tracer concentration of clarified effluent.

The trial data shown in Figure 4 are similar to the model prediction for zero removal efficiency, confirming the accuracy of the model and also confirming that the potassium tracer is not selectively removed in the system. At the conclusion of the trial, the model predicts that the clarifier outlet potassium concentration is at 78% of the final steady state value and that a total trial length of 400 minutes (6.7 hours) would be needed to reach 90% of the final steady state concentration.

Other Indicators

Chemical oxygen demand (COD) and turbidity of return clarified process water were used to indicate the cycle-up of other species. It should be noted that COD and turbidity are composite indicators of concentrations of multiple species. As previously discussed, each species will have a characteristic cycle-up time, which depends on its removal efficiency in the system. Figures 5 and 6 provide the results obtained from the same potassium tracer trial.

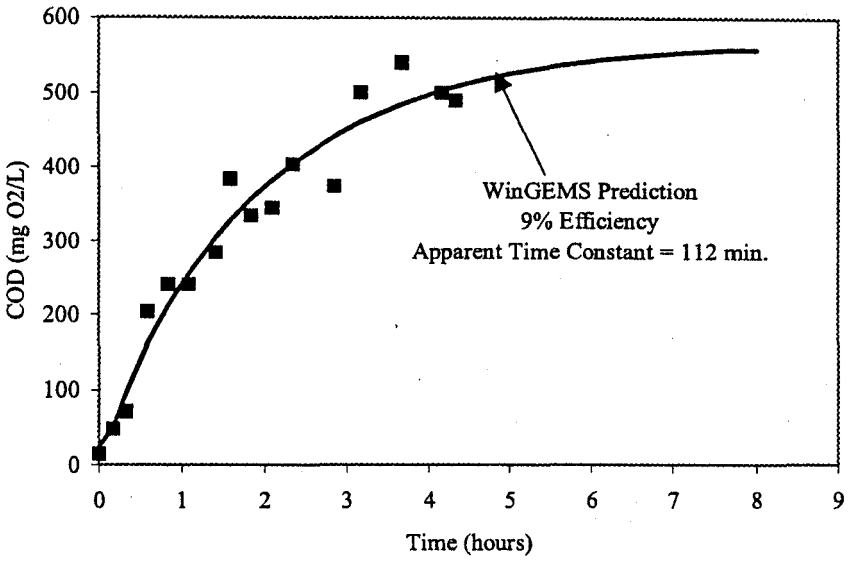

Figure 5. COD of filtered clarified effluent.

The COD data shown in Figure 5 are similar to the model prediction for 9% removal efficiency, which indicates that there are some species contributing to COD that is removed in the system.

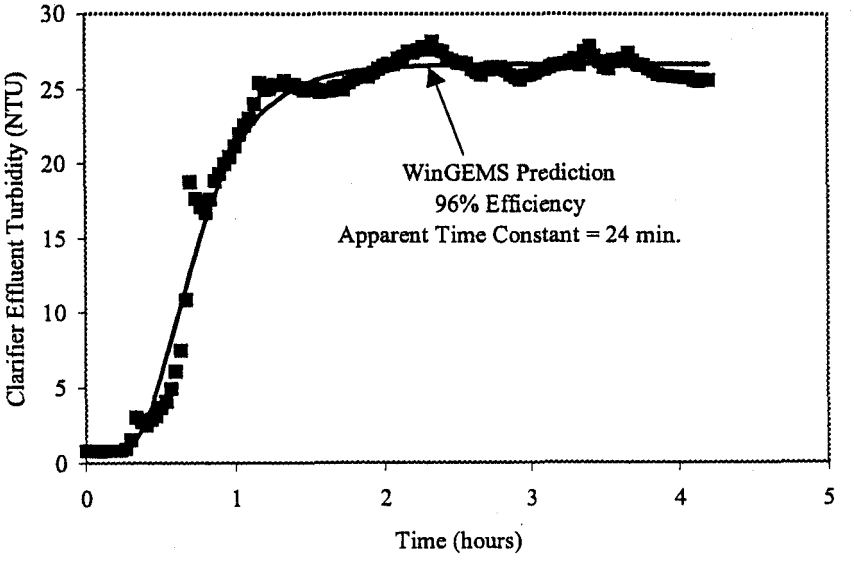

Figure 6. Turbidity of return clarified process water.

The turbidity results in Figure 6 show a much quicker cycle-up time and arrival to a steady condition as indicated by the close fit with the model prediction for 96% removal efficiency. The primary contributor

to turbidity in the system is ash, likely calcium carbonate. The clarifier is very efficient at removing ash, as exhibited by the rapid cycle-up of turbidity in the clarified process water loop.

The potassium tracer, COD, and turbidity results indicate that a model can accurately predict the behavior of the pilot system. With this model we are able to determine how to adjust the operating parameters to optimize fiber yield and contaminant removal. Although the pilot plant is simpler, better instrumented and better sampled than most mills, a validated model may be of great utility to recycling mill operators. For example, if the operators knew that a large quantity of contaminants were added to the pulper, they could predict when and how to change reject rates for various unit operations to minimize the impact of this potential upset. One could also use the model to change the operating parameters to best match the quality targets for a particular grade of product.

Conclusions

Use of modeling and tracer studies have been helpful in operating and optimizing the closed-loop recycling pilot plant. These tools have been used to define the trial run length that would be needed to reach steady state for the worst-case species in the system. Future plans will explore the impact of contaminant cycle-up on the behavior of adhesives during recycling. Furthermore, efforts will be directed at studying factors that control the generation of microstickies in the closed-water loop.

Acknowledgements

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