A Systematic and Fully Automated Procedure for Water and Element Balancing over Pulp and Paper Mills
A Case Study at Visy Tumut Mill

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ABSTRACT

A general strategy to perform water and element balances was developed and implemented for a pulp and paper mill to identify the flow profiles of water and key elements. The materials covered in the balance were water, sodium (Na), sulfur (S), calcium (Ca), potassium (K), and chlorine (Cl). A sampling strategy for the whole plant was first developed and then a number of analytical techniques were chosen, taking into account the special/difficult characteristics of the samples found in a typical Kraft pulp and paper mill. To perform the balances, boundaries around each section of the plant were selected and additional information was created by incorporating extra knowledge/relationships within each section, thus providing redundant information. Error analysis and data reconciliation were performed to evaluate the reliability of the results and correct them, to finally obtain an adjusted set of data consistent with the balance equations. The reconciled data obtained, as expected, showed better consistency in regards to the balance equations and reduced uncertainties. The final mass balance simulation toolkit developed enhanced the calculation and data processing activities, allowing material balances to be performed continuously to provide up-to-date results to serve for "on time" monitoring and "on time" decision making by mill staff.

I. INTRODUCTION

Environmental and economical sustainability is driving the pulp and paper industry towards ever increasing closure of the water and chemical cycles within the pulp and paper making process. A high degree of closure means that non-process elements, such as chlorine and potassium that enter in trace amounts in the wood and makeup chemicals, tend to accumulate within a mill. This can cause a number of problems such as corrosion, scaling, recovery boiler fouling and plugging [1], and deficient product quality. Further, restrictions on water usage and the utilization of treated process effluent for irrigation, means that water usage and effluent contaminant levels must also be carefully monitored. Therefore, a material balance study is often considered as a practical tool to provide water and chemical flow profiles for monitoring the closure in pulp and paper mills.

A material balance study was conducted over the Visy Tumut Pulp and Paper Mill (VTPPM), allowing tracking of flow profiles, and continuous monitoring of the mill's operational performance. The materials covered in the balance were water, and the key elements: sodium (Na), sulfur (S), calcium (Ca), potassium (K), and chlorine (Cl). The scope of this project encompassed a balance over all main functional areas, including those responsible for providing service utilities, such as cooling water.

The VTPPM employs the Kraft pulping process to produce strong paper suitable for linerboard packaging. The mill is a highly integrated process consisting of three main flow cycles: fiber processing, chemical recovery, and cooling water (Figure 1).

To complete the balance study, firstly a systematic procedure for sampling and sample analysis was developed to determine the amount of water, and key elements within the process streams of the mill. Secondly, a series of mass balances were constructed over all the areas previously mentioned. Thirdly, error analysis and data reconciliation techniques were applied, to evaluate the reliability of the results and compensate for experimental uncertainty, and obtain an adjusted data set more consistent with the balance equations. Finally, a material balance toolkit was developed to fully automate the overall procedure.

II. SAMPLE COLLECTION

In order to conduct the required mass balances it was necessary to collect and analyze the critical process materials within the mill.

The greatest possible accuracy to the mass balance could have been obtained by sampling every material within the mill. This however was not practical or necessary, and sampling was limited to materials within the mill containing significant and unknown quantities of the balance components. Very dilute streams and purchased chemicals were excluded.

With the materials requiring sampling identified, it was necessary to collect samples over a sufficient length of time, and with a sufficient frequency, to capture an accurate picture of the water and key elemental concentrations within the mill. The ideal period and frequency should provide sufficient resolution to capture small process fluctuations, such as those caused by the action of a control value maintaining a set point, and should do this over a sufficient length of time to capture gradual changes, such as those caused by changes in the source region for the mill's wood. In choosing the period and frequency it is was also necessary to consider the project time constraints (6 months) and resources available in addition to these factors, as sample analysis is a time consuming and costly procedure.

The ideal period and frequency is unfortunately best determined from experience, as each mill is different and there are many factors affecting concentrations fluctuations. It was generally accepted within the mill that a change in the liquor cycle takes three days to propagate, and that two weeks of continuous sampling is required to close a mass balance over an individual process area. Using this knowledge, samples were collected three times a week, for a period of four weeks, to provide a sufficient margin of error to capture major concentration changes in the mill, and provide enough time to close the balances.

This sampling program still provided a significant number of samples. To reduced the number within the project time constraints and resources, and yet still maintain an accurate representation, each weekly sample set was composited, to give four weekly sample sets.

III. SAMPLE ANALYSIS

The use of Inductively Coupled Plasma Emission Spectrometry (ICP) was chosen as the main means of analysis. ICP was chosen because of its ability to simultaneously analyze for a wide range of elements, including sulfur that is too electronegative to be analyzed by other methods [3]. ICP also offers relative freedom from chemical interference due to the long residence times and extreme temperatures experienced by a sample, resulting in almost complete atomization [4]. To increase accuracy, additional analysis of some samples containing calcium in very high or very low concentrations relative to the other elements, such as in the grits and dregs, was performed using Atomic Absorption Spectroscopy (AAS). AAS was used over ICP, as it is able to detect calcium over a much wider concentration range, and is a cheap and rapid way to determine the concentration of a single element.

Chlorine is too electronegative to be detected by any spectrographic method, and thus it was necessary to use an alternate method of analysis. A review of the analytical methods for chlorine analysis [4] showed that the most rapid and practical method for determining chlorine concentration is the use of an ion selective electrode (ISE). This method used to analyze the samples for total chlorine.

All of the analytical methods used require the sample to be in the form of an aqueous solution. For accurate analysis the samples must ideally consists of free ions in solution, unhindered by organic and colloidal material; this is essential for use of an ISE. With the exception of some water soluble samples, most samples collected required digestion to put them in this form. A number of methods and reagents are available to perform sample digestion, most of which involve heating a sample with a mixture of strong acids and oxidizing agents.

The need to analyze for chlorine and sulfur limited both the choice of method and reagents available, as both of these elements are the basis of many commonly used strong acids, and in addition these elements from volatile compounds during digestion. These limitations were overcome by performing the digestions using a mixture of nitric acid and hydrogen peroxide in microwave digester, which due to the use of sealed pressure vessels minimizes volatile loss. Typically 5mls of liquid samples or 0.5g of solid samples were digested with nitric acid (5ml, 69%) and hydrogen peroxide (1ml, 30%) using a mild digestion program (250W – 1min, 0W – 2min, 250W – 5min, 400W – 5min, 600W – 5min).

IV. MASS BALANCE CONSTRUCTION

Mass balances were performed over all parts of the mill, using a combination of the sample analysis results, historical process data from the mill's distributed control system, and chemical inventory records.

To perform these balances, it was necessary to choose boundaries over which to perform the input and output flow calculations. These boundaries can be made to arbitrarily contain any fraction of the total process, but generally the smaller each boundary, the greater the available information about the process and difficulty of the balance. A boundary was used for each individual processing area within the mill, and it was found that this provided sufficiently detailed information about the process, while allowing the balances to be solved with the process information available, and a minimal degree of difficulty.

The VTPPM features one of the world's most advanced distributed control systems (DCS). This DCS measures and records most of the important material flows and state variables within the mill. With the exception of some of the chemical inputs, the DCS was sufficiently dimensioned that any unmeasured material flows could be determined from other measured flows. To determine the unmeasured chemical inputs, chemical inventory records were used. For the purposes of a mass balance the inclusion of such an advanced control system is extremely valuable. Process data can be rapidly obtained for the entire operating history of the mill, making very representative averages obtainable.

To perform the balances, process data was collected from the DCS corresponding to the sampling period. This data was processed by calculating the numerical mean and the uncertainty within this mean from the variance within the data set. Typically, raw process data will contain outliers caused by process upsets and breakdowns, which in large numbers can skew the mean. The standard method to remove these outliers is to apply a confidence interval to the data, which relies on the data conforming to a known distribution. It was found that the data from the VTPPM did not conform to any standard distribution, and thus without rigorous statistical processing, confidence intervals could not be applied. This was acceptable considering the project aim was to create a mass balance representing the normal operation of the mill, which will include process upsets and breakdowns. When using variance to calculate the uncertainty in the mean of a measured process variable, it is necessary to assume that any uncertainty contributed from instrumental error is insignificant when compared to that from process fluctuations, which is reasonable given that typically the difference is several orders of magnitude.

V. RESULTS

The results of the water balance showed that water was mainly circulated within the paper machine, before being used for counter-current washing within the fibreline and then the digester.

Sodium, potassium and sulfur all behaved similarly as these elements where almost entirely circulated within the chemical recovery loop (i.e. from the white liquor to the weak black liquor and vice versa). The loss of sodium and sulfur (cooking chemicals) were accounted for in the amount of these elements remaining in the paper product, as well as in the losses from the chemical recovery process. The amount of these elements remaining in the paper is determined by the efficiency of the pulp washing process in the fibreline. The calcium balance results revealed that calcium, both dissolved and suspended, was carried from the paper machine into fibreline by the white water used for pulp washing in a counter-current flow fashion from the machine.

The uncertainties in the averages and analysis results were propagated through all of the mass balance calculations, allowing the results of the mass balances to be evaluated.

It was generally found that the discrepancies (residuals) within the component balances (water, Na, S, Ca, K, Cl) were close to or smaller than the uncertainty within the discrepancies, indicating that the discrepancies could have been negligible within the bounds of experimental uncertainty. This verified the accuracy of the balances in accounting for all of the material flows within the mill, and of the procedures for sample collection and analysis.

The only exception was the chlorine component balances. These showed discrepancies much larger than the uncertainties within the discrepancies, suggesting that a large systematic error had occurred within the balance procedures. Given that this systematic error was absent from the other component balances, the error could be traced back to method used to analyze the samples for chlorine. Chloride ion selective electrodes are very sensitive to sulfide ions, which at high concentrations cause the surface of the electrode to become passivated. At low concentrations the ions cause interference, and inaccurate readings. Even though measures were taken to destroy the sulfide in all samples known to contain sulfide (acidification and oxidization), these measures proved to be insufficient, as shown by the results of the chlorine component balances.

Samples of the results obtained from the balances can be found in Figure 2, Figure 3, and Table 1.

VI. DATA RECONCILIATION

It was found that the data measurements, both laboratory analysis measurements and process measurements, all contained a small but significant degree of uncertainty. These uncertainties will lead to some degree of inaccuracy when the balance results are used to monitor, control, or make any decision regarding the process. Hence, advanced data reconciliation (DR) was used to compensate for the uncertainties associated with the data measurements.

In the context of this balance study, the key results are the elemental flows obtained from the material balance equations which were a linear combination of the elemental flows. Therefore, a linear DR technique was utilized, and a batch processing method [2] was used to solve the DR problem. The data reconciliation problem was formulated to minimize the weighted least-squares estimation:

$$\begin{aligned} &\underset{x}{Min}\ J = (y-x)^T\ \Psi^{-1}\ (y-x) \\ &s.t.\ \ Ax = 0 \end{aligned}$$

Where **A** is a matrix (m × g) of known constants, and **x** is the vector of variables containing the measured process variables. In this case, all variables are redundant. The solution to this problem is obtained using the Lagrange multipliers method. The new estimate of the process variable ($\hat{x}$) can be obtained as:

$$\hat{x} = y - \Psi A_1^T (A_1 \Psi A_1^T)^{-1} A_1 y$$

The Microsoft Excel Spreadsheet Package was used along with its Visual Basic Macro Language to perform all calculations. The reconciled balance results showed that for the flow values, all the balances were now satisfied (i.e. balance discrepancies were equal or close to zero) (Figure 3 and Table 1). The reconciled results also showed that the associated uncertainties were reduced/minimized.

VII. MATERIAL BALANCE TOOLKIT

To make any decision on time, the mill requires up-to-date results. Therefore, the time taken for acquiring and processing the data to provide useful and reliable results needs to be reduced, which can be achieved with the aid of a fully automated procedure. This section will discuss the main steps involved in the construction of this mass balance toolkit.

At the VTPPM, process variables are measured by various measurement devices linked to the Distributed Control System (DCS), processed, and finally stored in the Information Management System (IMS). Each measured variable has a unique tag, consistent in both the DCS and the IMS. To retrieve the flow data from the IMS for a certain period of time, Simple Query Language (SQL) was used to communicate with the IMS and extract the required data for a specified tag and time period. The data retrieved was then imported into an Excel Spreadsheet with the aid of Visual Basic. Error analysis functions and data reconciliation formulation were also constructed in Visual Basic to process the propagated uncertainty calculations. A historical data base was also built into the toolkit to record and store key variables before each new update. This database is useful for monitoring the mill's operation, checking process consistency, and makes an excellent trouble shooting tool, as the source of process changes and upsets can be tracked.

The results obtained are reported using three main techniques which allow staff and management to obtain an informative overview of the mill's current operation. Reporting techniques include: (1) Graphical Interfaces (Figure 4), (2) Graphical Trends, and (3) Tabulation. The material balance simulation toolkit has made the mass balance fast and repeatable in a standard and systematical procedure. It also allows the mill staff to check the consistency of the mill's performance, as well as to perform trouble shooting by accessing the historical database.

VIII. CONCLUSIONS

The material balance strategy developed to determine the flow profiles of water and key elements within the VTPPM is repeatable, simple, labor saving, and rational, without compromising the level of accuracy. This procedure allows ongoing monitoring and management of water and chemicals within the VTPPM.

IX. REFERENCES

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[4] Skoog D. A., West, D. M., and Holler, F. J. (1996), “Fundamentals of Analytical Chemistry”, 7th edition, Sounders College Publishing,

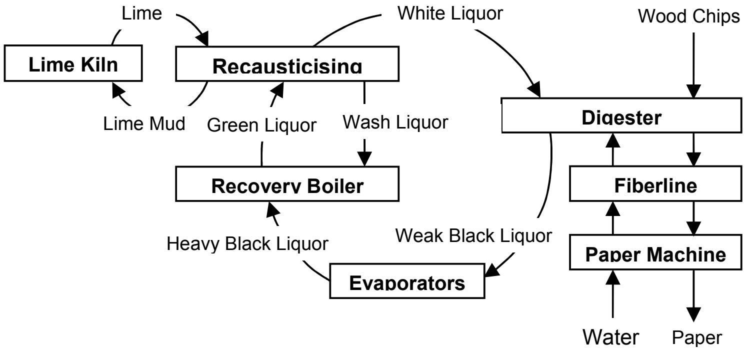


Figure 1: Schematic representation of the Kraft process within the mill.

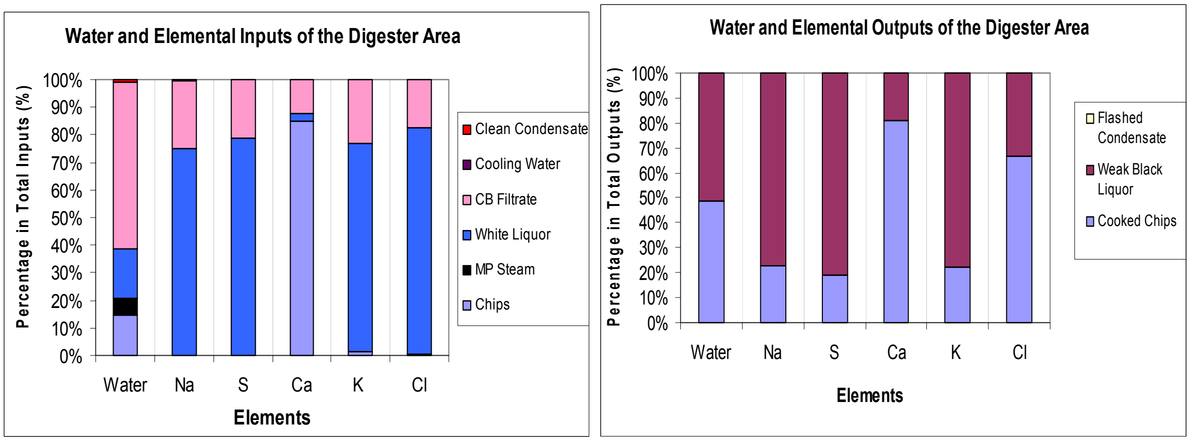


Figure 2: Percentages of water and elements input and output flows in the Digester Area.

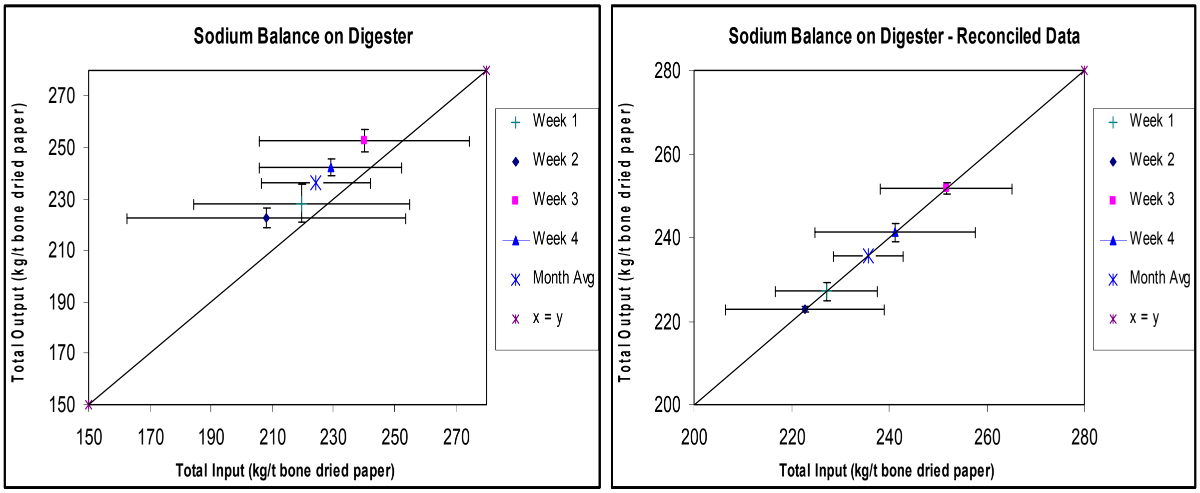
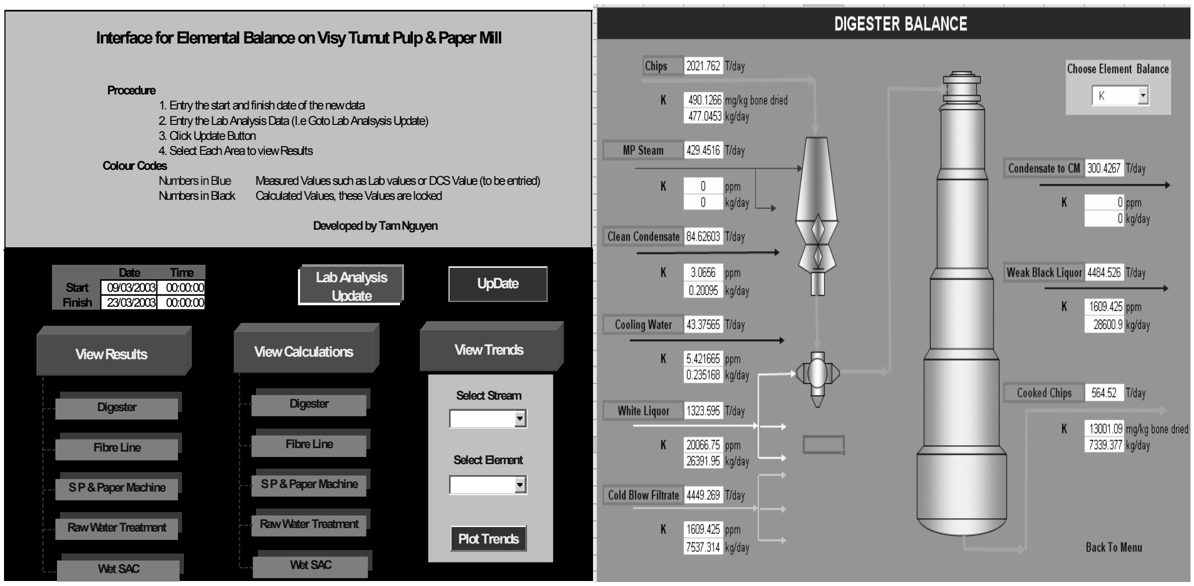


Figure 3: Elemental sodium balance results before and after data reconciliation in the Digester Area.

Table 1: Values of Na flows in different streams before and after the data reconciliation within the Fibre-Paper processing area.

Streams	Na Before		Na After	
	Average	Uncertainties	Average	Uncertainties
Chips	0.02	0.00	0.0226	0.0009
MP Steam	0.00	0.00	0.000	0.000
White Liquor	170	20	182	7
Cold Blow Filtrate	55	5	52	1
Cooling Water	0.004	0.006	0.005	0.001
Clean Condensate	1.18	0.00	1	0.00
Cooked Chips	53	1	53.9	0.3
Weak Black Liquor	183	2	182	1
Condensate to Cond Mill	0.00	0.00	0.000	0.00
Back Water	3.8	0.4	3.6	0.1
Seal Water	0.00	0.00	0.002	0.00
Pulp	5.2	0.5	5.5	0.1
Steam to HE	0.00	0.00	0.000	0.00
MP Steam to Steam Sys	0.00	0.00	0.000	0.00
Clean Condensate	0.01	0.00	0.015	0.004
White Water Condensate	0.03	0.00	0.034	0.001
Fresh Water	0.00	0.00	0.0024	0.0003
Recycle Paper	0.29	0.05	0.39	0.05
H_2SO_4	0.00	0.00	0.000	0.00
Alum	0.00	0.00	0.000	0.00
Paper	2.33	0.03	2.29	0.02
Condensate to Boiler	0.00	0.00	0.000	0.00
Steam Losses	0.000	0.00	0.000	0.00



Interface for Elemental Balance on Visy Tumut Pulp & Paper Mill

Procedure

1. Entry the start and finish date of the new data
2. Entry the Lab Analysis Data (I.e Goto Lab Analsysis Update)
3. Click Update Button
4. Select Each Area to view Results

Colour Codes

Numbers in Blue	Measured Values such as Lab values or DCS Value (to be entried)
Numbers in Black	Calculated Values, these Values are locked

Developed by Tam Nguyen

	Date	Time
Start	09/03/2003	00:00:00
Finish	23/03/2003	00:00:00

DIGESTER BALANCE

Chips 2021.762 T/day
K 490.1266 mg/kg bone dried
477.0453 kg/day
MP Steam 429.4516 T/day
K 0 ppm
0 kg/day
Clean Condensate 84.62603 T/day
K 3.0656 ppm
0.20095 kg/day
Cooling Water 43.37565 T/day
K 5.421665 ppm
0.235168 kg/day
White Liquor 1323.595 T/day
K 20066.75 ppm
26391.95 kg/day
Cold Blow Filtrate 4449.269 T/day
K 1609.425 ppm
7537.314 kg/day
Choose Element Balance: K
Condensate to CM 300.4267 T/day
K 0 ppm
0 kg/day
Weak Black Liquor 4484.526 T/day
K 1609.425 ppm
28600.9 kg/day
Cooked Chips 564.52 T/day
K 13001.09 mg/kg bone dried
7339.377 kg/day

Figure 4: Graphical Interface of the Menu.