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IMPROVING BROWN STOCK WASHING BY ON-LINE MEASUREMENT – MILL INVESTIGATIONS

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ABSTRACT

The aim of the study was to examine and improve the brown stock washing result and efficiency. It was done by utilizing real-time measurement devices and a monitoring system created for the purpose. Research work was carried out by installing 12 refractometers in the brown stock washing line at a Finnish kraft pulp mill. Refractometers continuously measure the dissolved solid contents of filtrate fractions. The refractometers and created monitoring system enabled the continuous evaluation of a washing result. The results indicated that the amount of washing loss had a clear influence on the performance of oxygen delignification, which was observed from the oxygen delignification's kappa reduction, chemical consumption and reactor's temperature. The results also indicated that by utilizing measurements it could be possible to control washers operation and then reduce washing losses from brown stock washing substantially. Results show that it is possible to evaluate the efficiency and the result of brown stock washing by utilizing continuous refractometer measurements and advanced data-analyzing tools. However, it is not simple and many variables should be noted if the aim is to develop and utilize a continuous monitoring system more efficiently.

INTRODUCTION

In chemical pulping, the primary reason for brown stock washing is to remove soluble impurities using a minimal amount of water (as this water must be evaporated later on). Pulp is also washed to recover valuable cooking chemicals and organic chemicals, which are recovered for their heating value /**1**/. Efficient washing improves the recovery of spent chemicals, reduces the consumption of reagents in the subsequent bleaching and limits effluent load from the plant /**2**/. It also has a positive effect on pulp quality and prevents deposition problems /**3**/.

The brown stock washing system is always mill dependent. It starts with cooking (Hi-Heat) and is followed, mainly in series, by various equipments such as a Drum Displacer (DD), a vacuum filter, a diffuser, a press filter, which use either dilution/thickening or displacement washing principles or their combination. The target is to connect these different washing equipments in a series and attain as good a washing result as possible with a minimum amount of used wash water /**1**/.

Brown stock washing also plays a key role in oxygen delignification performance. Washing before oxygen delignification is important, because a high incoming wash loss into the oxygen delignification reduces pulp strength and consumes oxygen and alkali. Washing after oxygen delignification is also very important, as it minimizes the amount of detrimental organic wash loss and cooking chemicals entering bleaching. When the amount of washing loss is high in the bleaching feed, more bleaching chemicals are consumed. With washing after oxygen delignification the economy and the environmental friendliness of the whole fibre line is improved /4/.

The parameters used to describe the performance of washing or its effectiveness can be divided into two categories: wash loss and dilution factor. Wash loss is defined as the amount of washable compounds in the pulp suspension that could have been removed in washing /**5**/. The dilution factor represents the net amount of water that is added during washing. The latter has been primarily used for running brown stock washing in the mills, since the water balance control should be kept in good condition to avoid unwanted spills from the liquor tanks. Nowadays the water balance controlling systems are more advanced in this respect, and more attention is paid to measuring wash loss.

Traditionally wash losses are evaluated by measuring individual parameters such as COD, TOC, sodium content, conductivity, etc.,

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and connecting them to mathematical calculations to measure mainly displacement ratio values (DR) or Norden efficiency factor, (E-values). However, the traditional parameters are not adequate enough to evaluate the real washing loss in real time **/5,6,7/**. The latest results **/8,9,10/** have shown that refractometer measurements can be used to solve this dilemma. The refractometer measures the concentration of washable liquid substances in real-time. It detects very well dissolved organic and inorganic materials, which have high refractive index (for example lignin, sodium, chloride and sulfate) which are also mainly responsible of wash loss in chemical pulping.

The experimental part studies the possibilities to improve and monitor the performance of a brown stock washing line by utilizing continuous refractometer measurements and advanced process data analyzing tools. One of the aims was to build a monitoring system, which can be utilized to analyze the result and efficiency of the washing line. The benefits of the monitoring system were studied by carrying out trial runs in different washers.

MATERIALS AND METHODS

Refractive index measurement principle

The refractometer measures analyze concentrations in solutions based on a measurement of the refractive index. Refractive index measurement is actually a measurement of the speed of light in a medium. The speed of light in a medium depends on the medium itself, temperature and wavelength. The refractive index depends on the concentration of dissolved solids. In general, the bigger the molecular size of the dissolved solids the bigger the refractive index per concentration unit is. The measurement accuracy is not influenced by particles, bubbles, fibres, color or temperature changes in the process medium.The laboratory reference temperature is usually 20°C or 25°C. Due to the wavelength dependency, the refractive index is measured with monochromatic light. The measurement principle behind the measurement of dissolved dry solids content through refraction has been presented in detail in our earlier studies ***/8,10/***.

Software tool used for process analysis

Wedge is a software commercial tool (developed by Savcor Forest Ltd.) designed for the management of process data and the analysis of process fluctuations. It consists of mathematical tools **/11/** which makes it possible to monitor and analyze process fluctuations systematically. One of the Wedge's advantages is that it consists of tools to overcome lag times. By utilizing Wedge it is possible to collect and analyze a mill's process data continuously and historically. If necessary, it is possible to remove faulty measurement periods from the process data **/11/**.

Installation of the Refractometers

The research was carried out by installing 12 continuous refractometers, which measured concentration levels, i.e. total dissolved solids of filtrates, in the brown stock washing line at a Finnish kraft pulp mill, **Figure 1**. Measurement devices were installed to allow the

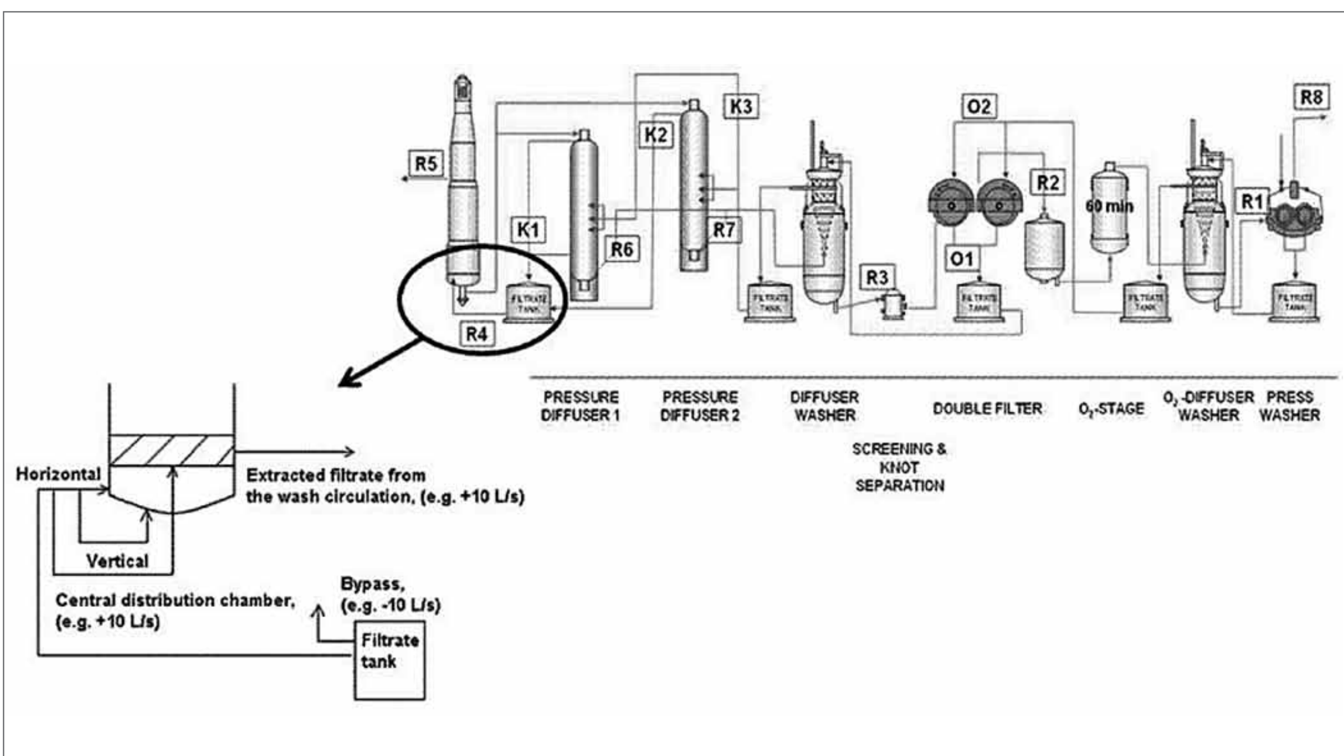

Figure 1. Installation sites of the refractometers at the pulp mill's brown stock washing line

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Table 1. Installation sites and the signs of the refractometers

Wash liquor and wash filtrate measurements		
Installation site	**Model**	**Sign**
Pressure diffuser 1, wash filtrate	PR-23	K1
Pressure diffuser 2, wash filtrate	PR-23	K2
Pressure diffusers 1 & 2, wash liquor	PR-23	K3
Double filter, wash filtrate	PR-23	O1
Double filter, wash liquor	PR-23	O2
Digester, wash liquor	PR-01	R4
Digester, extraction liquor	PR-01	R5
Liquor in pulp measurements (in-line refractometers)		
Installation site	**Model**	**Sign**
Wash press, pulp in	PR-01	R1
Double filter, pulp out	PR-01	R2
Screening, pulp in	PR-01	R3
Pressure diffuser 1, pulp out	PR-23	R6
Pressure diffuser 2, pulp out	PR-23	R7
Wash press, pulp out	PR-23	R8

evaluation of the efficiency and performance of the washing line. In addition, **Table 1** defines more precisely the installation sites.

Before trial runs, the refractometers were calibrated in cooperation with the refractometer supplier. Calibration was made by taking liquor samples from all the installation points. The samples were taken from both hardwood and softwood campaigns. From the pulp samples the liquor was squeezed out before analyzing, which was done about half an hour after sampling. The amounts of dissolved solids were analyzed by using SCAN-N 22:77. All the calibration analyses were made in the pulp mill's laboratory.

After the analyses the refractometers were calibrated to match the laboratory analysis results. Different wood species have different refractive index calibration curves. Despite that, different calibration curves between wood species were not used in this work. This is due to the fact that sufficiently accurate calibration was achieved without that procedure. Furthermore, campaigns between hardwood and softwood species are short at the experimental pulp mill, e.g. softwood campaigns often last less than a day. As a result, equilibrium in the filtrate tanks between different wood species is not clear.

The washing result analyses and calculation of different factors defining the performance of the washing line were carried out by utilizing Wedge. For instance, E-value calculations were created in Wedge. In addition, Wedge was utilized to calculate liquor balances

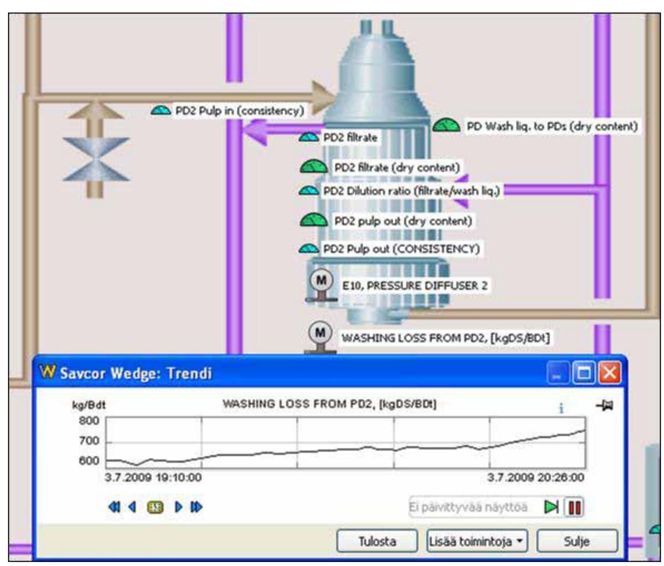

Figure 2. A screenshot from the flow sheet created in Wedge

and dilution factors in the washing line. Process data were collected in Wedge from the mill's data acquisition system. Two different time domains in the data acquisition were used, i.e. a minute average and an hour average. In **Figure 2** a screenshot from the flow sheet created in Wedge is represented.

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Calculations

The actual process E-value (APE-value) /**12**/ with fixed consistency, i.e. 10% consistency, is defined in equation 1

$$E_{10} = \frac{\ln\left[1+\frac{DF(y_1-y_2)}{L_1(x_1-y_2)}\right]}{\ln\left[1+\frac{DF}{9}\right]} \text{, where} \qquad (1)$$

L_1 = washed pulp stream, tons of liquor/o.d. ton of washed pulp

x_1 = concentration of dissolved solids in washed pulp stream, kg dissolved solids/ton of liquor

y_1 = concentration of dissolved solids in wash liquor stream, kg dissolved solids/ton of liquor

y_2 = concentration of dissolved solids in outlet liquor stream, kg dissolved solids/ton of liquor

The dilution factor (DF) can be calculated from equation 2

$$DF = V_2 - L_1 = V_1 - L_0 \text{, where} \qquad (2)$$

L_0 = unwashed pulp stream, tons of liquor/o.d. ton of washed pulp

V_2 = wash liquor stream, tons of liquor/o.d. ton of washed pulp

V_1 = filtrate stream, tons of liquor/o.d. ton of washed pulp

The washing loss can be calculated from equation 3

$$washing\,loss = x_1 L_1 \qquad (3)$$

The pressure diffuser's dilution ratio can be calculated from equation 4

$$Dilution\ ratio = \frac{wash\ filtrate, [l/s]}{wash\ liquor, [l/s]} \qquad (4)$$

Performance of the experiments

During the experimental part, the main raw materials used at the pulp mill were birch and pine. A minor part of the softwood raw material comprised spruce, i.e. around 15%. Below are represented the trial runs carried out around the digester washing and the pressure diffusers.

The main idea in the digester washing trials was to improve the utilization of wash water circulation in the digester. The purpose was to improve radial displacement washing which occurs on the wash circulation screen. This is done by increasing the amount of wash liquor fed into the digester and by extracting the same amount of filtrate from the wash circulation. The excess amount of wash liquor was introduced into the central distribution chamber. In addition, the amount of digester washing bypass was decreased simultaneously, (see Figure 1).

In this trial the wash water circulation was utilized for approximately 10 hours within one hardwood campaign. The amount of extracted filtrate from the wash circulation was about 0,7 t/BDt of pulp during the trial. Furthermore, the dilution factor of the digester washing was increased with the same amount. The digester's dilution factor before the trial run was around 0,2 t/BDt. There were neither big changes nor complications in the digester's runnability during the trial period or after the trial period. For instance, the blow consistency and the chip level were quite constant.

The effect of the downward velocity of the screen on the pressure diffusers' washing efficiency was studied by changing the set point of the screen velocity. The effect of the screen velocity was studied by slowing down the velocity stepwise. The experiments were carried out both with hardwood and softwood.

Furthermore, the effect of the dilution ratio on the pressure diffusers' washing efficiency was studied. This was done by increasing the set point of the dilution ratio.

RESULTS AND DISCUSSION

Washing in the digester

Figure 3 shows the washing loss from the digester and the amount of extraction from wash circulation. The field between the black lines indicates the period when liquor from wash circulation was extracted, i.e. the trial period.

As can be seen from Figure 3, the amount of washing loss decreased during the trial. However, more noticeable is that after the trial period the washing loss started to increase notably. As a result, it can be said that the digester's washing efficiency can be increased by enhanced radial displacement washing, which occurs in the wash circulation screen.

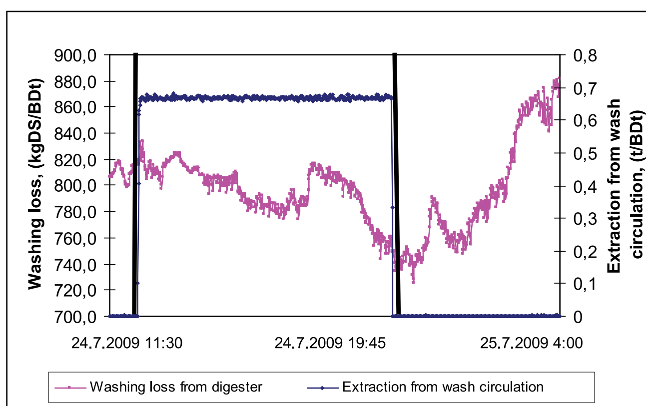

Figure 3. Washing loss from the digester and the amount of extraction from wash circulation. The field between the black lines indicates the trial period when the digester's dilution factor was increased and extraction from the wash circulation was utilized. Hardwood, 1035 BDt/d

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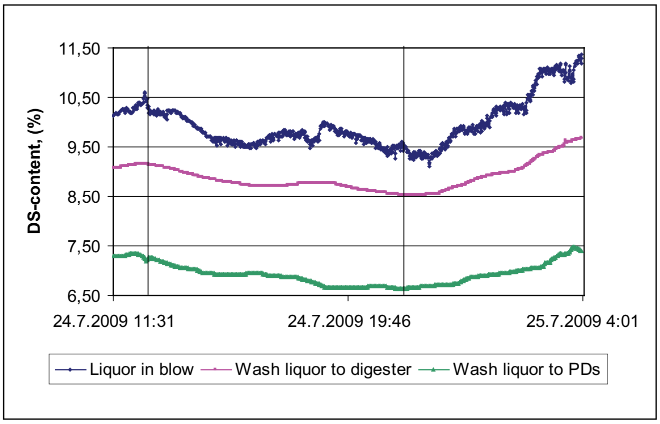

Figure 4. **Dissolved solid contents of blow pulp suspension, wash liquor to the digester and the pressure diffusers. The field between the black lines indicates the trial period when the digester's dilution factor was increased and the extraction from wash circulation was utilized. Production rate was 1035 BDt/d, hardwood**

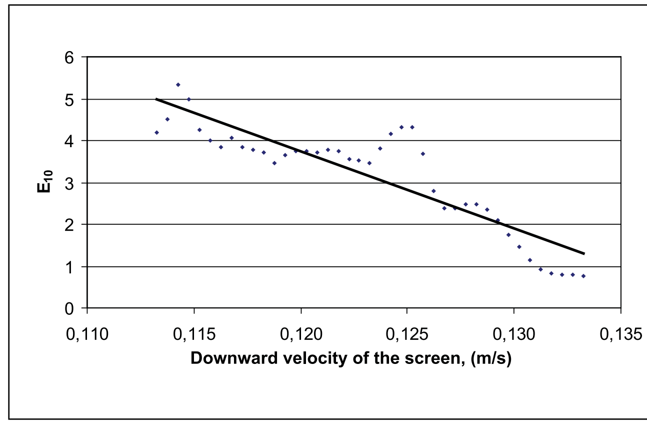

Figure 6. **The effect of the downward velocity of the screen on the pressure diffuser's washing efficiency. Hardwood, the production rate to pressure diffuser 1 was 520 BDt/d**

Figure 4 represents the dissolved solid contents of liquor with blow pulp, wash liquor to the digester and wash liquor to the pressure diffusers from the same trial run.

As can be seen from Figure 4, the concentrations of different liquor fractions decreased during the trial. On the contrary, the concentrations started to increase after the end of the trial. This supports that the trial run had a favorable effect on the digester's washing result.

Washing before oxygen delignification

Figure 5 represents the effect of the pressure diffusers feed consistency on the washing efficiency.

As can be observed from Figure 5, with a higher feed consistency the washing efficiency increased. The results attained were similar to what we **/10/** obtained in our mill investigations with a pressure filter. However, in this example the pressure diffuser's washing efficiency was unsatisfactory even if the feed consistency was high. This is due to the fact that other variables affecting the washing performance were not correctly adjusted.

Figure 6 represents the effect of the downward velocity of the pressure diffuser screen on the washing efficiency. Furthermore, **Figure 7** demonstrates the effect of the downward velocity of the pressure diffuser screen on the washing loss from the same trial run.

From Figures 6 and 7 one can notice that the downward velocity of the screen notably affects the pressure diffusers washing result. In this trial run the E_{10}-value increased linearly from around 1 to 5 when the downward velocity of the screen was slowed down. At the same time, the washing loss from the pressure diffuser decreased notably. From the results reached, it can be stated that when the downward velocity of the screen is adjusted to too high a level the washing efficiency of the pressure diffuser deteriorates.

The optimum downward velocity of the screen always depends on the velocity of the pulp. The velocity ratio between the screen and the pulp should be close to 1. If the velocity of the screen is too fast compared to that of the pulp flow, it leads to the formation of a

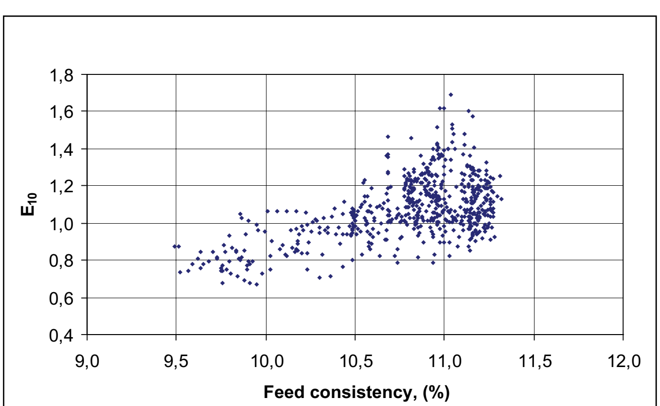

Figure 5. **The effect of the feed consistency on the pressure diffuser's washing efficiency. Hardwood, 1080 BDt/d**

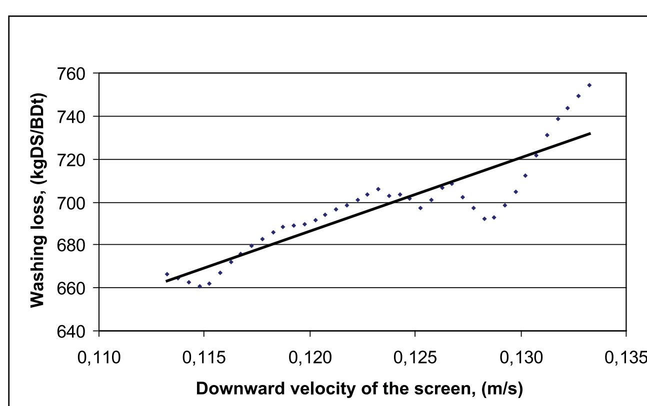

Figure 7. **The effect of the downward velocity of the screen on the pressure diffuser's washing loss**

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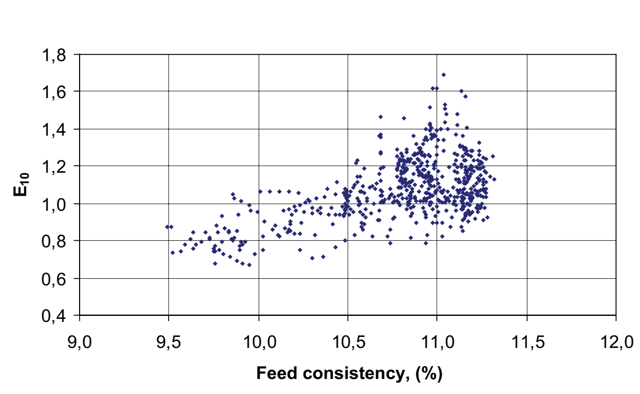

Figure 8. The effect of the dilution ratio on the pressure diffuser's washing efficiency. Softwood, the production rate to pressure diffuser 1 was 450 BDt/d

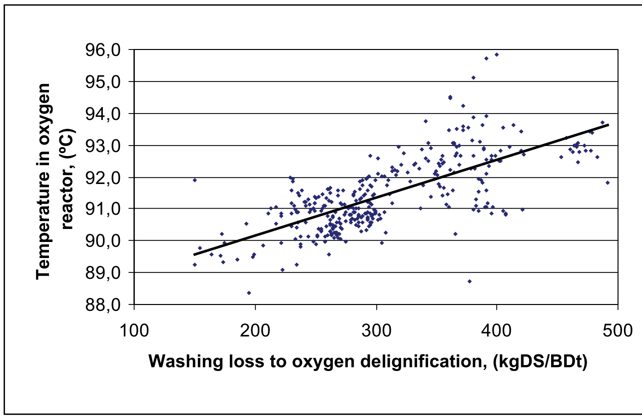

Figure 10. The effect of the washing loss on the oxygen reactor temperature with softwood. An hour average from June 2009 to August 2009

thinner pulp web. By increasing the velocity difference, the risk of a web breakage also increases. However, it is also possible to adjust the downward velocity of the screen too slow, which can cause plugging of the screen. The pressure in the web increases resulting in local dewatering when the velocity of the screens downward movement is lower than that of the inlet pulp **/13/.** The results attained are similar to those obtained by Lysen **/13/** in his calculation from mill data with pressure diffuser. Lysen has demonstrated that the optimum velocity ratio is slightly higher than one being from 1.1 to 1.3.

Figure 8 demonstrates the effect of the pressure diffuser's dilution ratio on the washing efficiency.

From Figure 8 it can be seen that by increasing the dilution ratio the washing efficiency can be increased. The reason for this is that the dilution ratio set point affects the dilution factor value. Even if the amount of wash liquor, i.e. V_2, is kept the same, the dilution factor increases when the dilution ratio set point is increased. This is due to the fact that discharge consistency and the amount of wash filtrate increase since the dilution ratio is increased.

Figure 9 represents the effect of the washing loss on the oxygen delignifications response.

As can be observed from Figure 9, the oxygen delignification response was highly affected by the amount of washing loss

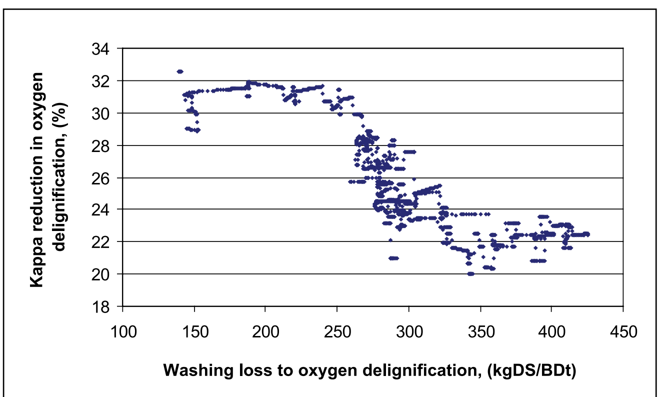

Figure 9. The effect of the washing loss on the oxygen delignification's kappa reduction. Hardwood, production rate from 1035 to 1125 BDt/d

with hardwood. When the amount of washing loss in the oxygen delignification feed increases the kappa reduction decreases. This is due to the fact that oxygen is consumed in the oxidation reactions of the washing loss. In other words, the selectivity and performance of the oxygen delignification decrease as the amount of washing loss increases. The biggest deterioration of the oxygen delignification's kappa reduction was when wash loss increased from a value of 250 to 300 kgDS/BDt. The wash loss should therefore be less than 250 kgDS/BDt to guarantee unimpeded oxygen delignification performance in this experimental mill.

Miller *et al.,* **/14/** obtained quite similar results to us. They have shown that the retardation of delignification is approximately one kappa unit when the total solids carry-over to the oxygen stage is increased by 50 kgDS/BDt.

Figure 10 represents the effect of the washing loss on the oxygen reactor's temperature with softwood.

From Figure 10 it can be noticed that the temperature in the oxygen reactor correlated clearly with the amount of washing loss. As the washing loss to oxygen delignification increased, the temperature in the oxygen reactor increased. The temperature in the oxygen delignification tower can increase and thus accelerate unselective reactions in the fibre because of the residual alkali present in the black liquor and the exothermal oxidation reactions **/15/.** In addition, the high washing loss level may increase the amount of hydroxyl radicals, which has a negative effect on the cellulose degradation and viscosity loss **/5/**.

CONCLUSIONS

It is possible to evaluate the efficiency and the result of brown stock washing by utilizing continuous refractometer measurements and advanced data-analyzing tools. However, it is not simple and many variables (production rate, flow rate and consistency measurements, time-delays, etc.) should be noted if the aim is to develop and utilize a continuous monitoring system more efficiently. The results indicated that the efficiency of the digester washing can be

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increased by utilizing a wash circulation and the extraction from the wash circulation more efficiently. The results also indicated that the washing efficiency of the pressure diffusers depends outstandingly on the downward velocity of the screen. By controlling the screen velocity correctly the washing efficiency can be increased considerably. In addition, the set point of the dilution ratio has a great influence on the pressure diffusers' washing performance. Also, the oxygen delignification response is highly affected by the amount of the washing loss, which was observed from the oxygen delignification's kappa reduction, chemical consumption and reactor's temperature. However, by utilizing refractometers and data-analyzing tools it is possible to discover the black spots in the washing line and evaluate a washing result continuously. In other words, it enables the improvement of the washer's efficiency and reduce the wash loss level to oxygen delignification and to bleaching. In the future it is recommendable to utilize refractometers in the washing line and bring higher level process optimization, e.g. at the whole washing department. Good positions to measure the washing loss are at least at the blow pulp, pulp after a pre-oxygen washing and pulp after a post-oxygen washing.

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REFERENCES

1. Crotogino R.H., Poirier N.A., Trinh D.T. (1987), *The principles of pulp washing*, Tappi Journal, 70(6):95-103.
2. Tervola P., Gullichsen J. (2007), *Confidence limits in mass balances with application to calculation of pulp washing efficiency*, Appita Journal, 60(6):474-481.
3. Wilson J. (1993), *Results from improved brown stock washing*, TAPPI Pulping Conference, Proceedings, pp155-157.
4. Andbacka, S., (1998), *The importance of washing in oxygen delignification and TCF bleaching*, Pulp & Paper Canada, 99(3):57-60.
5. Sankari, M. (2004), *Real wash loss compounds in Kraft pulp delignification and bleaching,* Doctoral thesis, University of Oulu, Department of Chemistry, Oulu, Finland, p.53.
6. Sillanpää, M. (2005), *Studies on washing in Kraft pulp bleaching*, Doctoral thesis, University of Oulu, Department of Process and Environmental Engineering, Oulu, Finland, p.41.
7. Bottan, G. (1993), *Is COD test a wash-out on washing*? Pulp Pap. Intl.35(3):.51
8. Kopra R., Tirri, T., Dahl, O. (2008), *Refractive index measurements for brown stock washing loss – laboratory investigations*, Appita Journal, 61(5): 408-412.
9. Kopra R., Helttunen J., Tervola P., Tirri T. and Dahl O. (2010), *Refractive index measurements for brown stock washing loss - mill investigations*, Appita Journal 62(2):131136.
10. Kopra R., Karjalainen S., Tirri T. and Dahl O. (2010), *Optimization of pressure filter performance by refractometer - Mill investigations,* 64th Appita Annual Conference Proceeding pp 207-214, Melbourne, Victoria, Australia, 19.-22.4.2010.
11. Anon. (2007), *Savcor Wedge V.6.1 User's Manual*. Savcor Forest Oy, p 273.
12. Haapamäki, P.O. (1979), *Modified Norden's method for pulp washing calculations*, Chemical Workshop, Montreal, p.26.
13. Lysen C. (1987), *Control of screen velocity – The key to pressure diffuser washing efficiency*, Pulp washing symposium 1987, Proceedings pp 321-333 Maarianhamina, Finland, May 18-21, 1987, The Finnish Pulp and Paper Research Institute, The Finnish Paper Engineers' Association.
14. Miller W. J., Shackford L. D., Jiang H. & Genco J. (1991), *Effect of entrained black liquor carry-over on medium-consistency oxygen delignification*, Tappi Journal, 74(2):117-122.
15. Vuorenvirta K., Fuhrmann A., Gullichsen J. (2000), *Effect of black liquor carry-over on selectivity of oxygen delignification*, International Pulp Bleaching Conference, Proceedings pp.65-70, Halifax, Canada.