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# USING A PROCESS REFRACTOMETER FOR MEASURING THE PERFORMANCE OF FIBERLINE PROCESSES

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## SUMMARY

In chemical pulping, the purpose of brown stock washing (BSW) is to remove both organic and inorganic soluble matter from the pulp suspension using the lowest possible amount of wash liquor in the fiber line. Without effective washing, the optimal perform of oxygen delignification and the optimal economic results of chemical pulp production cannot be reached.

Brown stock washing was a popular topic in the 1980s and 1990s. At the same time, the oxygen stages became more common on fiber lines. There is currently renewed interest within the pulp industry in $O_2$ delignification, brown stock washing, and real-time control of washing, because of environmental, energy, economic and legislative issues.

Due to the better yield attained in chemical pulping, there is a trend toward pulp being cooked for higher kappa and the residual lignin being removed by post-delignification in the $O_2$ stage. In addition, BSW has a great effect on the performance of the oxygen stage. An increase in washing loss causes reductions in pulp viscosity, strength, and yield in the $O_2$ stage. Correspondingly, efficient washing is key to low-cost bleaching. So on-line measurements which indicate wash loss levels as a dissolved material are needed.

This study demonstrates how an on-line process refractometer can be used in brown stock washing to monitor wash loss levels and washing performance and to calculate washing equipment efficiency. This work also shows interactions between brownstock washing and the oxygen delignification. Furthermore, it also discusses the economic benefits which can be achieved in the fiber line using real-time measurement emphasizing especially the energy savings.

**Keywords:** Brownstock washing, Energy saving, On-line measurements, Oxygen delignification, Process refractometer.

## INTRODUCTION

In chemical pulping, the purpose of brown stock washing derived from the cooking stage is to remove both organic and inorganic soluble matter from pulp suspension using the lowest possible amount of wash liquor. Efficient washing improves the recovery of cooking chemicals (Na and S) and wood-based dissolved organic material. It also reduces the additional consumption of reagents in the subsequent bleaching stages [1]. Sufficient washing also has a positive effect on pulp quality and it prevents deposition problems efficiently [2]. The main target of oxygen delignification is to continue delignification that started in cooking in a more selective manner than occurs in the digester (i.e., remove a substantial fraction of the residual lignin using oxygen and alkali at a moderate temperature). Delignification with oxygen is a gentler way of reducing the kappa number than extended cooking. Lowering inlet kappa to bleaching also decreases bleaching chemicals consumption and, because of this, reduces organic wastewater load from bleaching.

Washing result is described by the amount of wash loss/carry-over. Wash loss is the amount of soluble (washable) inorganic and organic compounds that still remain in the pulp suspension after washing. Wash loss can originate from the pulp itself (reaction products) or from the wash water used (e.g. condensates, recirculated washing filtrates). The wash loss denotes the amount of a substance which escapes with the pulp leaving the washing system. Frequently, the wash loss is expressed as chemical oxygen demand (COD) or saltcake loss. The latter has been used traditionally because it was easy to analyze and provided an indication of the recovery rate of inorganic chemicals. Whilst interest in the recovery of chemicals persists for economical reasons, the focus has been shifting towards organic compounds as closed cycles and environmental aspects gain increasing importance in process design [3]. The latest study [4] suggest measuring an amount of total dissolved solids (TDS), which represents both organic and inorganic load, using process refractometer and it can be measured in real time both from the filtrates and the filtrate fraction of the pulp. Additionally, by measuring the three or four incoming/outgoing dissolved dry solids streams to the washers together with consistencies and flow rate measurements, it is possible to calculate the real-time effectiveness of the washer(s).

Wash loss substantially affects the performance of many sub-processes in the fibre line, including oxygen delignification. High wash loss into the oxygen delignification stage due to poor brown stock washing reduced the performance of oxygen delignification and decreased both pulp strength and consumed oxygen and alkali [5-9]. Thus, the anticipated kappa reductions are not achieved [7]. The performance of oxygen delignification also significantly affects bleaching costs. When the Kappa number after the oxygen stage is reduced, less chemicals are needed in the following bleach plant. During washing after the oxygen stage, it is also important that dissolved reaction products from oxygen delignification be washed away before bleaching and the desired chemicals savings are achieved. By using copious quantities of washing water, it is possible to decrease wash loss and reduce bleaching chemical costs. Improved brown stock washing indirectly decreases the costs of waste water treatment plant (WWTP) in a chemical pulp mill. If more black liquor -based organics and salt cake that can be kept in the liquor cycle, less material is sent to the WWTP. These savings include a reduction in nutrient chemical costs and could possibly also lead to a decrease in the number of aerators required to operate in the mill's WWTP aerated stabilisation basin [10].

The brownstock washing therefore has an impact on the operation and economy of the entire pulp mill. Without effective washing the economic viability of chemical pulp production is affected. To make washing as efficient as possible, parameters that affect the operation of a washing system could be controlled and found optimal ways to operate them. Parameters include process conditions such as the dilution factor, feed and discharge consistencies, pH, temperature, or entrained air. They also include equipment-specific parameters, such as a particular traveling speed, mechanical pressure, or fluid pressure or vacuum [3]. The operation of the fiber line should be monitored as versatile as possible with various online measurements. This work presents the utilization of a process refractometer to measure TDS concentration changes on the washers and in the oxygen stage.

## EXPERIMENTAL

### *Refractive index measurement principle*

The refractometer measures the concentration of washable liquid substances in real time. It detects dissolved organic and inorganic materials which are also mainly responsible for wash loss in chemical pulping, very well due to their high refractive indexes (for example lignin, sodium, chloride and sulphate). The refractometer measures liquid concentration 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, fibers, 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, figure 1. In most applications the measurement area is kept clean by the self-cleaning effect of the prism surface. In fibre mass, lines the constant abrasion caused by the fibres keeps the prism surface clean from coatings of any kind. On the filtrate side fouling is caused, mainly by extracts of the birch mass. In such cases, to ensure the reliability of the measurement, a regular steam wash is used to clean the prism surface. The device monitors cleanliness with self-diagnostics. The measurement principle behind the measurement of dissolved dry solids content through refraction has been presented in detail in our earlier studies [4,11,12].

**Figure 1. Refractive index digital measurement principle and sensor.**

### *Measurement arrangements*

Studies have been carried out both on individual washers, on the entire brownstock washing line and also in the oxygen stage. In this work, the focus is mainly on individual washers and the oxygen stage. Figure 2 shows a conventional measurement arrangement with a single DD-washer, where the concentration of the feed pulp, the washing liquid, the vacuum filtrate (describes the discharge pulp quite well) and the dissolved solids of the outgoing filtrate are measured. Figure 3 shows measurement arrangement how we can use process refractometers for studying the performance of oxygen delignification. Also Echowise gas measurement was used in the feed of $O_2$ DD-washer. Based on these measurements, we gathered information about the behavior of dissolved matter and gas content to the washer in the pulp mill’s oxygen delignification stage.

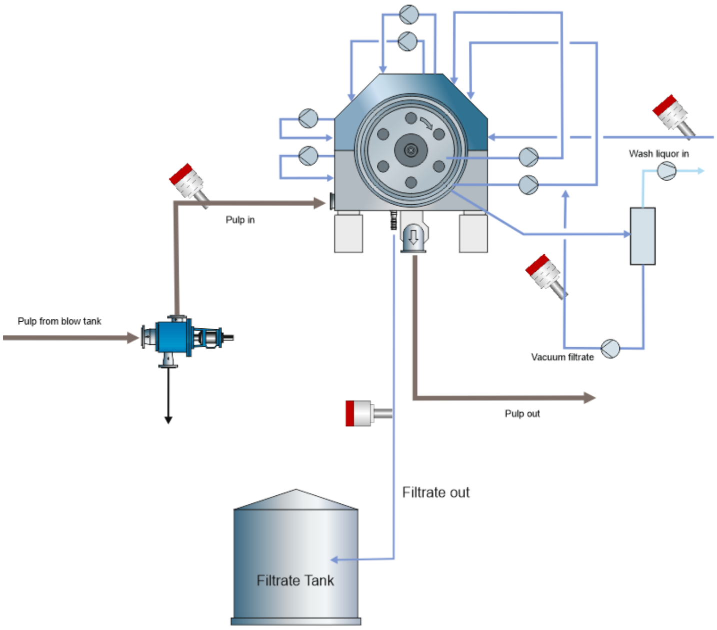

**Figure 2. Measurement arrangements at the four-stage DD-washer of the Scandinavian pulp mill fiber line**

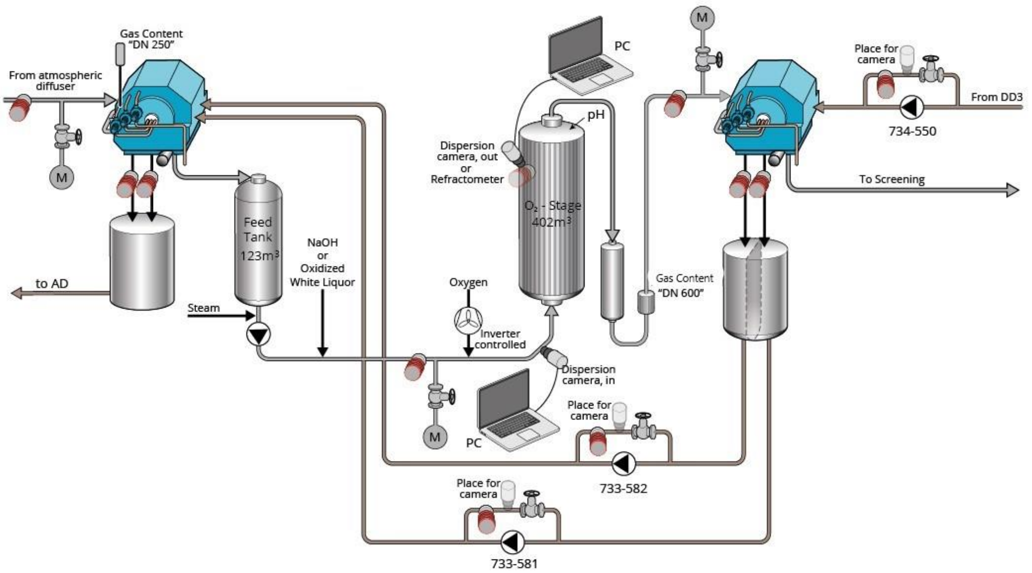

**Figure 3. Measurement arrangements of hardwood pulp fiber line (650,000 a.d. metric tons/year) $O_2$ delignification**

Measurements have been used to obtain data on the operation of the washing and the oxygen stage. In addition to that, we have carried out step wise tests in which optimal feed consistencies have been sought. Also the effects of wash water amount on efficiency of washing have been investigated. By measuring the TDS content from pulp suspensions and washing liquor, it is possible to measure washing results in real time. In addition, by measuring the three or four incoming/outgoing TDS streams to the washers together with the consistencies and flowrate measurements, it is possible to calculate the real-time effectiveness of the washer(s). Based on the concentration measurements and the mill's data collection system, efficiency values (DR and $Y_{10}$) has been calculated. Calculation has been shown detail in earlier studies [4,13,14]. Based on the calculated efficiency values, it has been possible to conclude which operating parameters are optimal at any given time.

## RESULTS AND DISCUSSION

### *Washers' optimization*

The experimental results of the dilution factor (DF) effect on the displacement ratio of the DD-washer are shown in **Fig. 4**. Increasing the DF increases the displacement ratio and thus improves washing results. On one hand the results indicate that increasing the DF from 1 to 3 can slightly increase the displacement ratio (DR). On the other hand, when the DF decreases below unity, the DR of the washer decreases very quickly, yielding a low washer performance. The more effective the washing, the bigger the change in DR velocity when DF is decreased. The DF should therefore be above unity to guarantee unimpeded washer performance. However, the use of a dilution factor above two is not feasible, because the higher amount of wash water would increase the cost of black liquor evaporation. Typically mills operate with a DF above 2 to reduce their bleaching costs, but in our examined mill the operating with a DF above 2 was not economically viable.

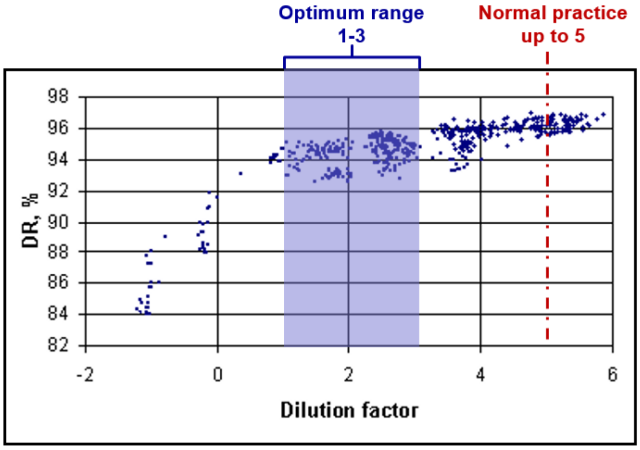

**Figure 4. Effect of dilution factor (DF) on the displacement ratio (DR) (Softwood).**

The experimental results of how washing consistency (the computational consistency on the washing locker) affects the $Y_{10}$-yield and displacement ratio are shown in **Fig. 5**. The Y-yield and displacement ratio increases when the washing consistency is increased. The reasons for that are the longer washing time and the growing wash ratio. When the average washing consistency is higher in the washer and the production ratio is kept constant, the drum turns slower and washing time increase. Also, when DF is constant and the washing consistency increases, the wash ratio ($W = V_2/L_1$) increases. This means that the pulp cake (fibres) obtain relatively more wash water. Clearly a higher consistency of feed pulp increases the displacement ratio and thus leads to a more effective economical pulp washing.

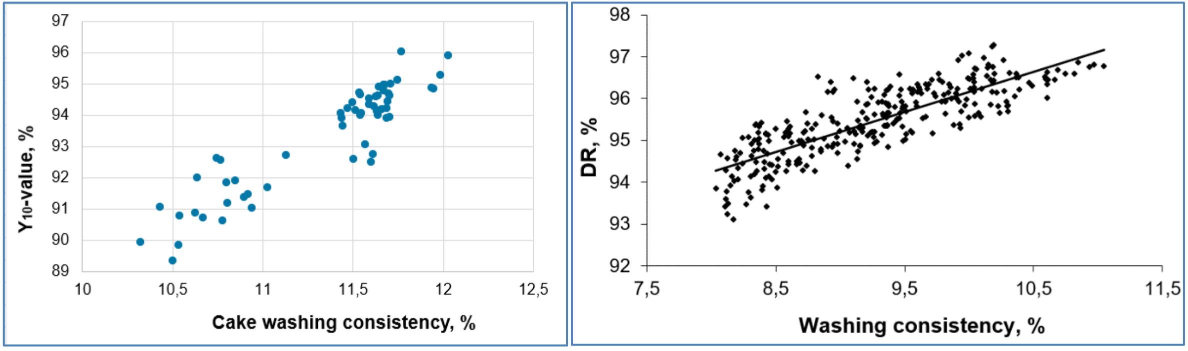

**Figure 5 a. Effect of the washing consistency on the DD washer's washing yield and b. on the displacement ratio at the two different mills. (DF approx. 2)**

### *Oxygen delignification*

The effect of the washing loss on the response of oxygen delignification is shown in **Fig. 6**. 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. Also more alkali is consumed in oxygen delignification when the amount of washing loss increases. The reason for this is that alkali is consumed in the neutralization reactions of the acids originating from cooking, cf. [7]. In other words, the selectivity and performance of the oxygen delignification stage decrease as the amount of washing loss increases.

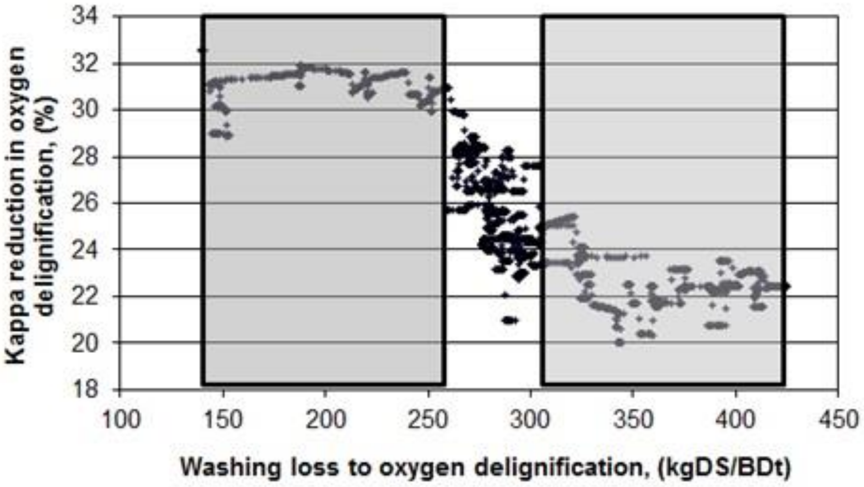

**Figure 6. The effect of the washing loss on oxygen delignification's kappa reduction, hardwood. Feed kappa to oxygen delignification was quite stable at 19 - 19.8, oxygen kappa varied from 12.7 to 15.2.**

The effect of the washing loss on the oxygen reactor's temperature with softwood is shown in **Fig. 7**. It can be seen that the temperature in the oxygen reactor correlates clearly with the amount of washing loss. As the washing loss to oxygen delignification increases, the temperature in the oxygen reactor increases. In the high wash loss levels, residual alkali present in the black liquor and the exothermic oxidation reactions of black liquor's sulphur compounds can increase the temperature in the oxygen delignification tower at the beginning of the reaction step and thus accelerate unselective reactions in the fibre [15].

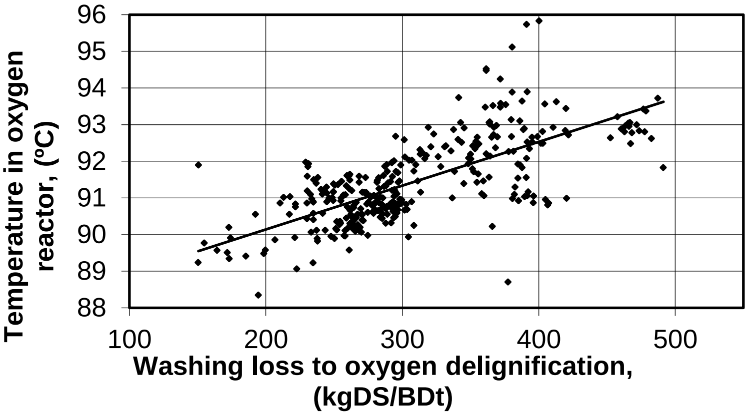

**Figure 7. The effect of the washing loss on the oxygen reactor's temperature with softwood.**

Figure 8 shows the gas content and temperature of $O_2$ DD-washers feed pulp. It can be seen that when temperature is lower, more gas goes to the washer. After the oxygen delignification, gases are removed by flashing at over 100 degrees, if the temperature is too low in flashing, the gases are not removed sufficiently, and may will end up to the next washer.

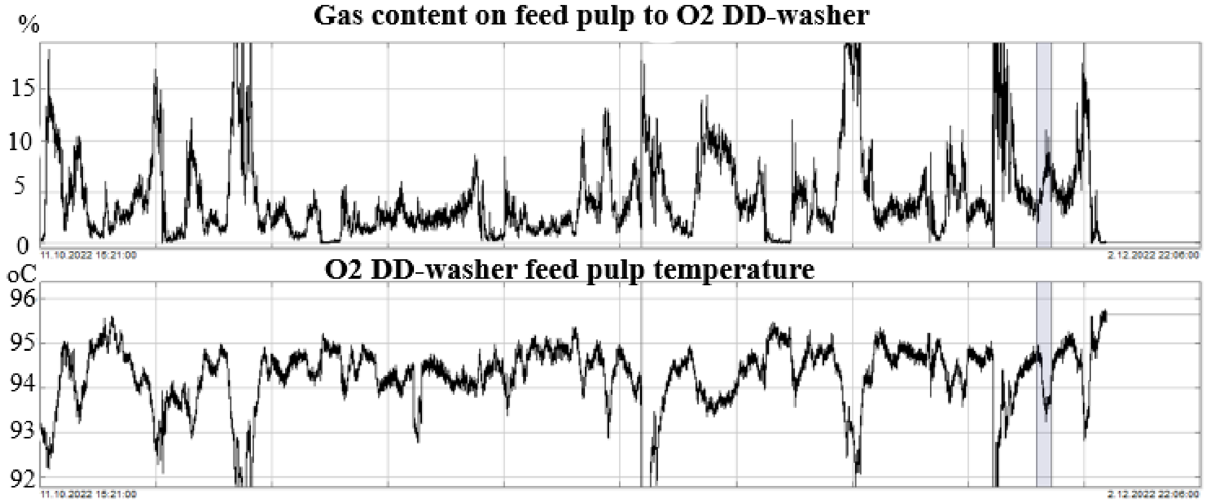

**Figure 8. Gas content and temperature in the feed of $O_2$ DD-washer measured with Echowise gas measurement.**

### *Energy savings*

In economic terms, the most important effects of the dilution factor are on the bleaching chemical costs, the evaporation costs and losses of salt cake [10]. Figure 9 shows that by increasing the dilution factor by one unit from Value 1 to Value 2, evaporation costs increase on average by €1.5/BDt in this experimental mill calculated with the values reported by the mill. This would make about 900,000€/a at 600 000 tons per year mill. Compton 1997 [10] study found that DF change from value 1 to value 5 increased costs from 8.5 to 14.5$/BDt, i.e. about 1.5$ per one DF unit. At the end of 2022, the price of electricity was very high due to the world political situation. In this case, the effect of evaporation costs on the overall economy of the pulp mill was even greater.

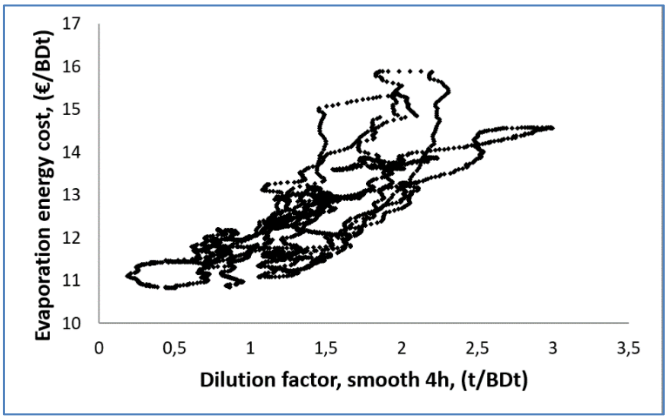

**Figure 9. Effect of dilution factor on the evaporation energy costs**

## CONCLUSIONS

The results indicated that it is possible to evaluate the efficiency and the result of brown stock washing by utilizing continuous process refractometer measurements and mill's data collection system. 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.

The results indicated that amount of dilution factor and the feed consistency notably affects the performance of the DD-washer. 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. The water consumption has also a great influence on the economy of the fiberline, such as evaporation and chemical costs.

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 reduces the wash loss level to oxygen delignification and to bleaching and also enables the control of wash water usage to washers.

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## REFERENCES

1. Tervola, P. and Gullichsen, J., Confidence limits in mass balances with application to calculation of pulp washing efficiency. Appita Journal, 60(6):474-481, (2007).
2. Wilson J. (1993) Results from improved brown stock washing. Proc. of 1993 Tappi Pulping Conference, Atlanta, GA, USA, 155-157.
3. Krotscheck, A.W., Pulp Washing, in Sixta, H. (ed.) Handbook of Pulp, WILEY-VCH Verlag GmbH &Co. KGaA, Weinheim, German, (2006).
4. Kopra, R. Application of the Refractometer in the Measurement and Monitoring of Brown Stock Washing. Doctoral Thesis, Aalto University, Finland, 70 p, 2015.
5. Allison R., Suckling I., Sun Y. (2000) Dissolved solids entrainment in oxygen delignification: effect of specific components. Proc. of the International Pulp Bleaching Conference, Halifax, Canada, 71-80.
6. Iijima J.F., Taneda H. (1997) The Effect of Carryover on Medium-Consistency Oxygen Delignification of Hardwood Kraft Pulp. Journal of Pulp and Paper Science, 23(12):561-564.
7. Miller W., Sullivan D.P., Genco J.M., Zou H., Johnson D. and Kwon H.B. (1998) Medium consistency oxygen delignification design using a two phase concept. Part 2. High solids carry-over. Proc. of 1998 Pulping Conference, Montreal, Quebec, Canada, 229-242.
8. Miller W., Shackford L., Jiang H. and Genco J. (1991) Effect of entrained black liquor carry-over on medium consistency oxygen delignification. Tappi J. 74(2):117-122.
9. Andbacka S. (1998) The importance of washing in oxygen delignification and TCF bleaching. Pulp & Paper Canada 99(3):57-60.
10. Compton R. (1997) Economic analysis of brown stock washing systems. Pulping Conference, San Fransisco, CA, USA, 257-272.
11. Kopra, R., Tirri, T. and Dahl, O., *Appita Journal*, 61(5): 408(2008).
12. Kopra, R., Karjalainen, S., Tirri, T., et al., *Appita Journal*, 64(1): 49(2011).
13. Tervola, P. An equivalent wash yield and extension of the equivalent displacement ratio in chemical pulp washing. Appita 68(3), 258-266, 2015.
14. Kopra, R., Karjalainen, S. and Tervola, P., DD-washer performance optimization using the latest measurement technology, Proc. of Tappi PEERS 2022 Conference, Providence, RI, USA (2022)
15. Vuorenvirta K., Fuhrmann A., Gullichsen J., Effect of black liquor carry-over on selectivity of oxygen delignification. Proc. of International Pulp Bleaching Conference, Halifax, Canada, 65-70 (2000).