

**Results of the PhD Thesis on Real Time Refractive Index Measurement  
in Washing Process Optimization and Control**  
**Riku Kopra, MAMK**



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refractive index measurement in washing  
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## Biography

### **Engineer of Paper technology, Tampere University of Applied sciences, 2000**

- Practical training -97, Metsä-Rauma

### **Master of Science in Industrial Environmental Technology, University of Oulu 2004**

- Master thesis: Study of pulp washing in five different kraft pulp mill

### **Researcher: LUT Savonlinna 9/2004 – 10/2005, MAMK Savonlinna 11/2005 →**

### **Postgraduate, Aalto University 2006 →**

## Publications

### **Journal Papers**

- 1) Kopra R., Tirri, T., Dahl, O. (2008) Refractive index measurements for brown stock washing loss – laboratory investigations, Appita Journal, 61(5): 408-412.
    - Usability of Refractometer to Measure Washing Loss in Chemical Pulping
  - 2) Kopra R., Helttunen J., Tervola P., Tirri T. and Dahl O. (2010) Refractive index measurements for brown stock washing loss - mill investigations, Appita Journal 63(2):131-136.
    - Different measurement methods in determining brown stock washing loss were compared, Washing efficiency E10 was calculated, refractometer measurement in the oxygen delignification feed was tested.
  - 3) Kopra R., Karjalainen S., Tirri T. and Dahl O. (2011) Optimization of pressure filter performance using refractometer - Mill investigations, Appita Journal 65(1):49-54, 94.
    - Optimization of different variables which affect to pressure filters performance, refractometer measurement in the all filtrate fractions of single washer.
  - 4) Kopra R., Kari E., Harinen M., Tirri T. and Dahl O. (2011) Optimization of wash water usage in brown stock washing, TAPPI JOURNAL vol.10 no.9
    - Utilization of Real-time Refractometer Measuring Results in Chemical Pulping
  - 5) Kopra R., Kari E., Harinen M., Tirri T. and Dahl O. (2012) Improving brown stock washing by using on-line measurement – mill investigations, O PAPEL vol.73, num.1, pp.79-85.
    - Optimisation of brown stock washing, digester washing, pressure diffuser washing, double filter washing, the effect of the washing to oxygen delignification
- 2) Based on Jukka Helttunen's, 3) based on Simo Karjalainen's and 4) and 5) Erkki Kari's master thesis.
- 6) Kopra R., Toivakainen S., Tolonen P., Tirri T., Dahl O. (2013) Monitoring of activated sludge plant streams using online refractometers and conductivity analysers, O PAPEL vol.74, num.9, pp.61-65.

## Publications

- **Conference Papers**

- Kopra R., Helttunen J., Tervola P., Tirri T. and Dahl O., Refractive index measurements for brown stock washing loss - mill investigations, 63<sup>rd</sup> Appita Annual Conference & Exhibition, Melbourne, Australia, 19-22.4.2009, p.51-56.
- Kopra R., Karjalainen S., Tirri T. and Dahl O., Optimization of pressure filter performance by refractometer - Mill investigations, 64<sup>th</sup> Appita Annual Conference & Exhibition, Melbourne, Australia, 18-21.4.2010, p.207-214.
- Kopra R., Kari E., Harinen M., Tirri T. and Dahl O., Improving brown stock washing by using on-line measurement – mill investigations, ABTCP-TAPPI 2010 43<sup>rd</sup> Pulp and Paper International Congress & Exhibition, Sao Paulo, Brasilia, 4-6.10.2010, p.10.
- Kopra R., Kari E., Harinen M., Tirri T. and Dahl O., Optimization of wash water usage in brown stock washing, TAPPI PEERS Conference, Norfolk, VA, USA, 17-20.10.2010, p.12.
- Kivenheimo M., Kamrat E., Kopra R., Sellutehtaan ruskean massan pesun optimoinnilla suuria säästöjä, Automaatio XIX seminaari, 15.–16.3.2011, Helsinki, Finland.
- Kopra R., Tolonen P., Tirri, T., Dahl, O. Refractometer and its ability to measure dissolved dry solids at low concentrations, 65<sup>th</sup> Appita Annual Conference & Exhibition, Rotorua New Zealand, 10-13.4.2011, pp.179-189.
- Kopra R., Tirri T., Dahl O., Use of refractometer Measurement in Chemical Pulp Mill Applications, Asian Paper 2012 Conference, 25.-27.4.2012, Bangkok, Thailand.
- Kopra R., Toivakainen S., Tolonen P., Tirri T., Laukkanen T., Dahl O. Studies on wastewater treatment plant performance measurements, ABTCP 2013 46<sup>th</sup> Pulp and Paper International Congress & Exhibition, Sao Paulo, Brasilia, 8-10.10.2013.

## Background

- Brown stock and bleaching washing studies in last decades in the University of Oulu → Sankari's\* and Sillanpää's\* academic dissertations.
- Need for new way to measure wash loss
- At the same time K-Patents started their own studies in the Veitsiluoto pulp mill
- We started to search the suitable equipment suppliers.
- K-Patents and Honeywell
- 2006 started academic dissertation entitled Use of refractometer measurement for monitoring brown stock washing

\* Sankari Minna: Real wash loss compounds in kraft pulp delignification and bleaching, 2004

\* Sillanpää Mervi: Studies on washing in kraft pulp bleaching, 2005

## Wash loss measurements in sodium based cooking methods

### Traditional:

- Is defined as the amount of sodium, which leaves the washer room with the pulp.
- Did not take into account other substances retained in the pulp suspension

### Conductivity:

- Conductivity analyzers measures ionic concentration of electrolyte samples
- Correlation between filtrate conductivity and the amount of sodium gives on-line information from the need of Make-up chemicals

### COD:

- Is an indirect sum parameter of all compounds which are oxidized under defined reaction
- Off-line method, which gives information for wash loss load to bleaching and so information of bleaching chemical consumption ( should remember that some compounds can cause a lot of COD with no effect on the responses).

### Dissolved solids:

- Describes all dissolved material, inorganic and organic. Lot of dissolved lignin in BSW.
- Can be measured on-line by refractometer

### TOC

- Total organic carbon is the amount of carbon bound in an organic compound.
- Describes pretty well wash loss, but off-line method

### BTG lignin analyser

- Describes important wash loss compound lignin
- Do not work in all conditions and concentrations

→ We choose refractometer which measures all dissolved material as sum parameter

## Reasons for our choice

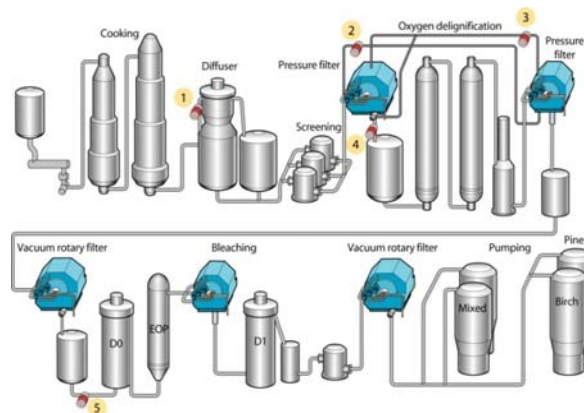
- A challenge has been found measuring technique, that measure “real wash loss” **sufficient reliable** and **continuous** in extremely **demanding conditions** (high alkalinity, pressure, the temperature) **in real-time with short response time**.
- In addition, the measuring technique should work **both in the liquid and in the pulp stream**, that on-line calculation of the effectiveness, which demands three of four incoming / outgoing dissolved dry solids streams to the washers, could be possible.
- There have been two approaches to measure the wash loss from the pulp stream; measure wash loss from the fibers or from the liquid part of the stream. Because over 50% of the wood material **is dissolved into the process liquor** in the delignification reactions, in the medium consistency (MC) pulp **90% of the pulp suspension is water** and in the alkaline brown stock washing **lignin is in dissolved form**, we have chosen the way to measure **dissolved dry solids in the liquid phase**.

## Hypothesis of the dissertation

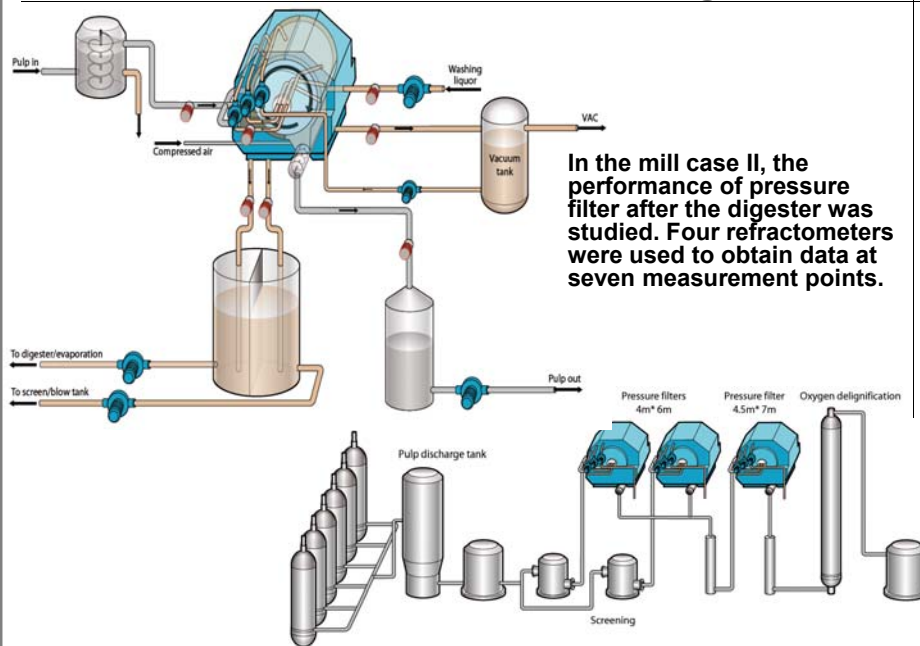
- Using refractometer as a measurement device ***it is possible to measure*** in pulp suspensions and in liquor streams dissolved dry solids with adequate accuracy.
- Measuring the dissolved dry solids from pulp suspension and from washing liquor it is possible to measure washing results in a real-time. Additionally, by measuring the three of 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 to the washer.
- Developing measuring system ***it is possible to improve*** the results of brown stock washing, reduce water consumption and thus improve energy economy of the whole pulp mill.

## Arrangement 1

In the mill study I, the focus was investigate brown stock washing performance before and after oxygen delignification in a kraft pulp mill. The research was performed by installing 5 refractometers on the brown stock washing line

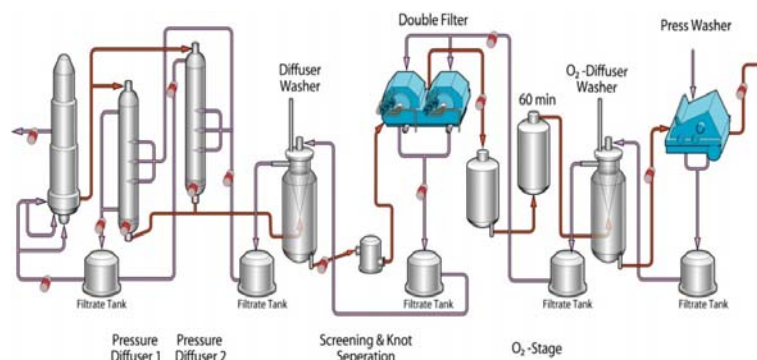


## Arrangement 2



## Arrangement 3

In the mill case III; the efficiency and performance of the washing line was studied. This was done by using real-time refractometer measurements and advanced process data analysis tools. 12 continuous refractometers were used to obtain data at 13 measurement points.

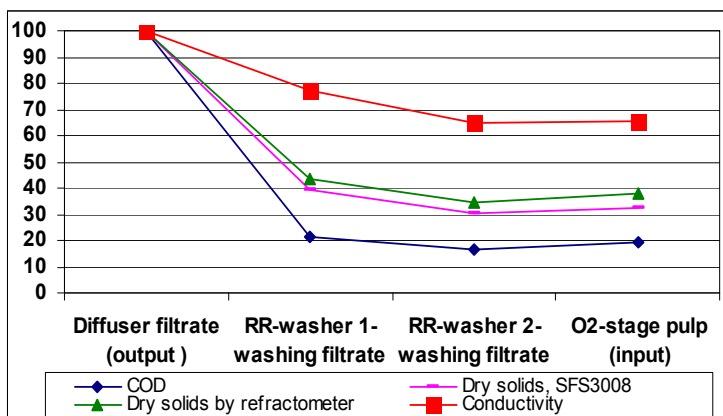


## Results

- Comparison of different measurement methods

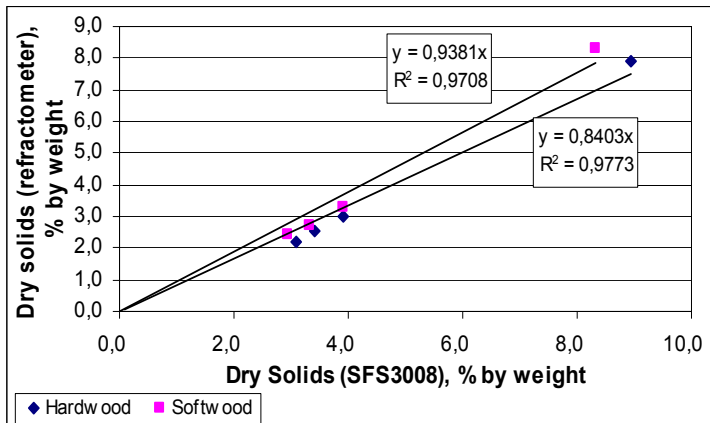
## Normalized wash losses

- All devices detect wash loss
- Proportional variances are constant



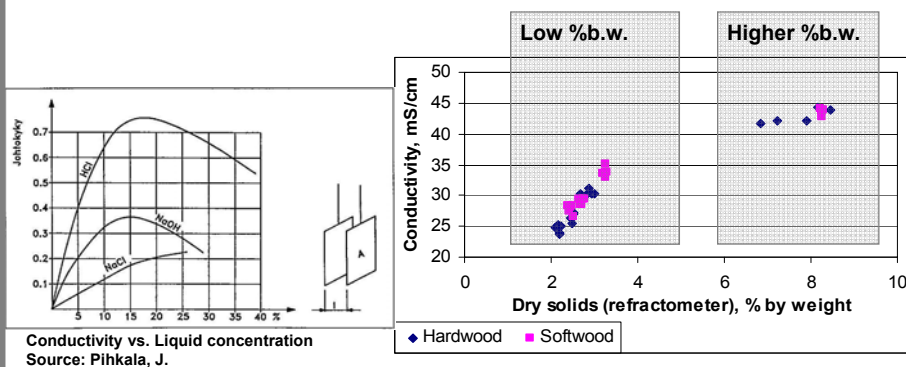
## SF-3008 vs. Refractometer in filtrates

Correctly calibrated refractometer measures exactly DDS.  
The wood species had resonance for the measuring result



## Conductivity vs. refractometer in filtrates

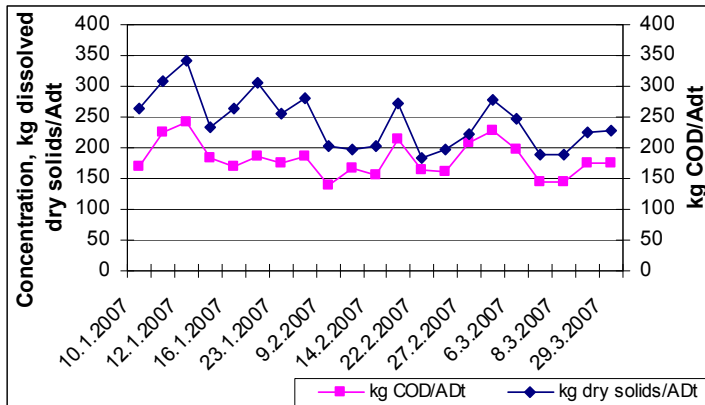
- The correlation is not linear
- Conductivity increase begins to slow down with a rise in dry solids concentration





## Filtered results of Refractometer and COD-analysis

- Refractometer measurement follows the mills COD-laboratory analysis considering level differences

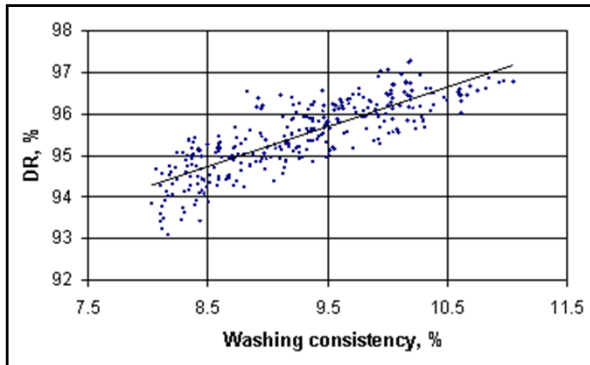


## Results

- The effect of pulp consistency to washers operation

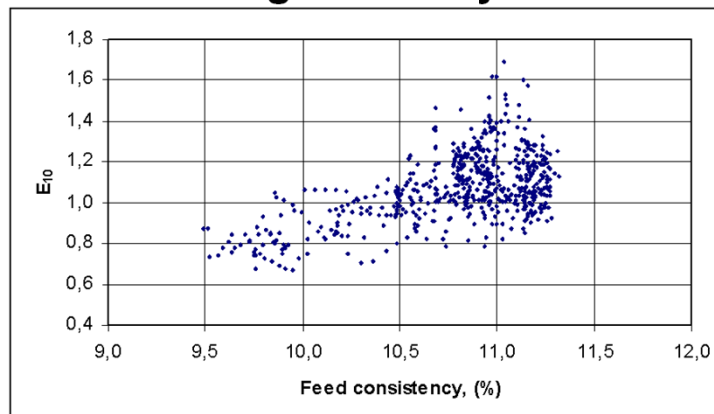
## Effect of washing consistency on washing results

- A sufficiently high consistency of feed pulp to the washer enabled high washing consistency → is an essential requirement for effective and economical washing of pulp



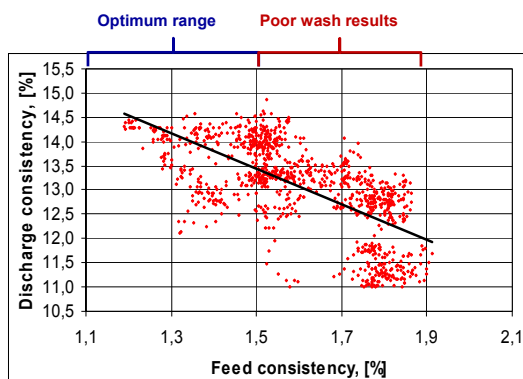
## Pressure Diffuser's Feed Consistency Effect

- With a higher feed consistency, the washing efficiency is increased



## Effect of Feed Consistency on Discharge Consistency (Hardwood)

- Too high feed consistency harmfully affects the double filter's washing performance
- In the literature: "When the washer operates at design capacity the optimum feed consistency is between 1.0 and 1.25% for softwood and 1.1 and 1.5% for hardwood"

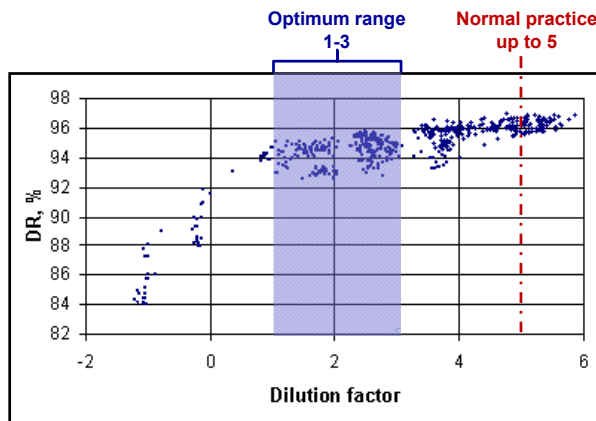


## Results

- The effect of wash liquor amount to washers operation

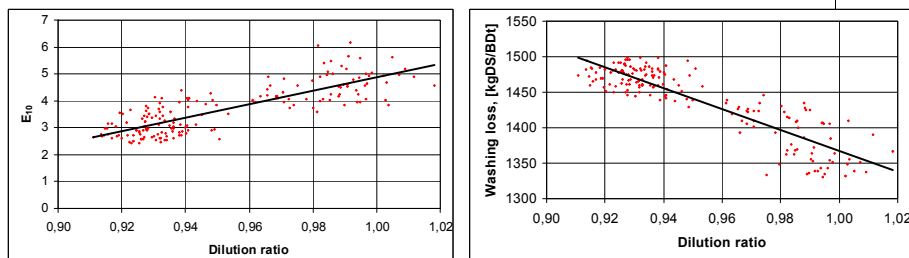
## Effect of dilution factor on washing results

- Dilution factor should be above 1 to guarantee unobstructed washer performance
- The real-time results are similar to literature



## Effect of Dilution Ratio on E10 and on Washing Loss

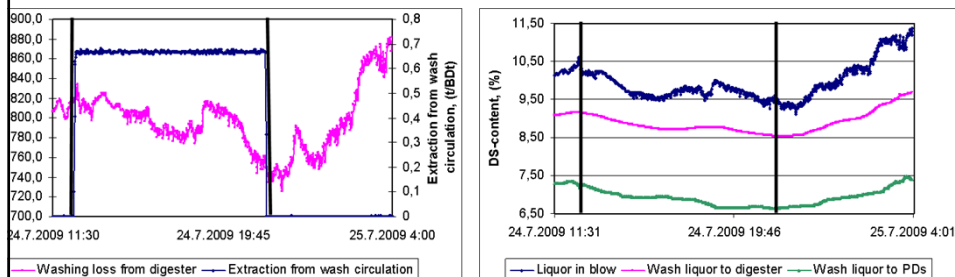
- The dilution ratio notably affects the pressure diffuser's washing result



$$\text{Dilution ratio} = \frac{\text{wash filtrate(out), [l / s]}}{\text{wash liquor(in), [l / s]}}$$

## Utilising radial displacement washing in the digester

- Bypass was decreased and the amount of wash liquor fed into the digester was increased (into the central distribution chamber). The same amount of filtrate from the wash circulation was extracted.
- The digester's washing result can be increased by utilising radial displacement washing.

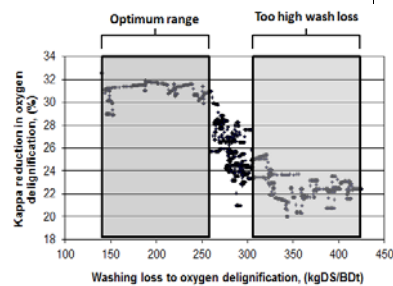
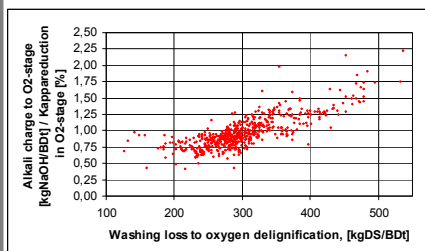


## Results

**The effect of the brown stock washing on the performance of oxygen delignification and on the economy of pulp mill**

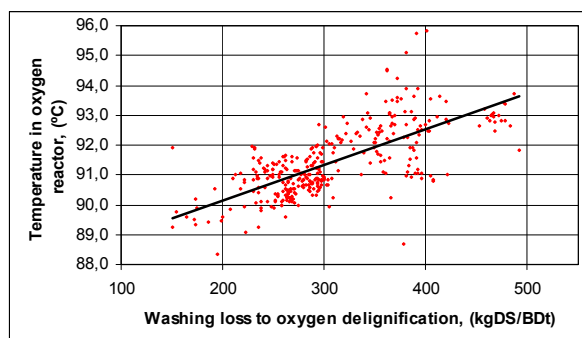
## Effects of Washing Loss on Oxygen Delignification (Hardwood)

- When the amount of the washing loss increases more alkali is consumed in oxygen delignification
- The reason for this is that e.g. alkali is consumed in the neutralization reactions of the acids originated from the cooking



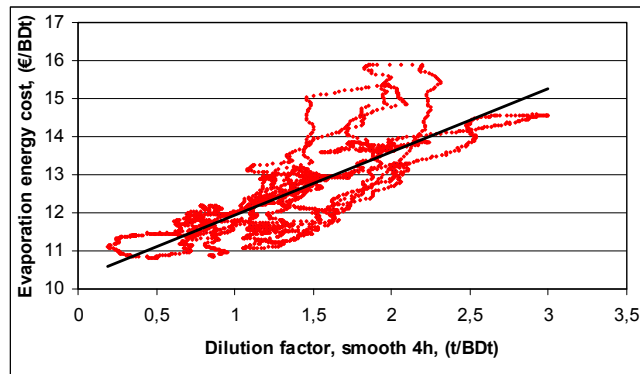
## The Effect of Washing Loss on Oxygen Reactor's Temperature (Softwood)

- The temperature in the oxygen reactor correlates with the amount of washing loss



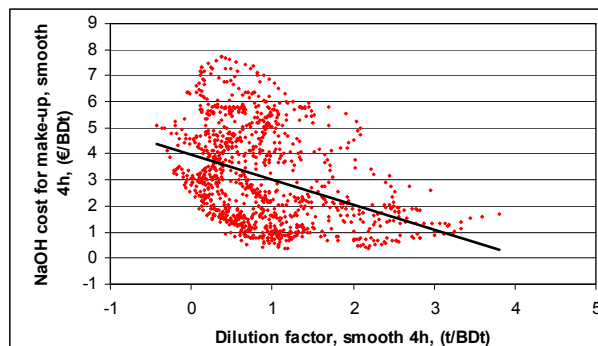
### The Effect of Dilution Factor on Evaporation Energy Costs

- At this mill experiment increasing the dilution factor by 1 unit, evaporation costs increase on an average by 1.5 €/BDt.



### The Effect of Dilution Factor on NaOH Make-Up Costs

- At this mill experiment increasing the dilution factor by 1 unit, the make-up chemical cost as NaOH decreases on an average by 1.0 €/BDt.



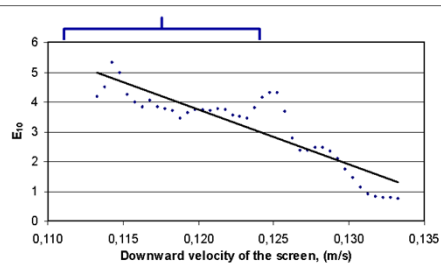
## Results

- Washer operation

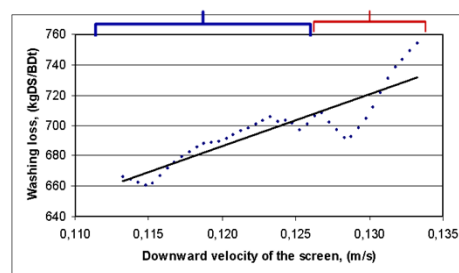
### Effect of the Downward Velocity of the Pressure Diffuser Screen

- The downward velocity of the screen notably affects the pressure diffuser's washing result
- In literature: "The optimum velocity ratio should be slightly higher than one being from 1.1 to 1.3."

Optimum range



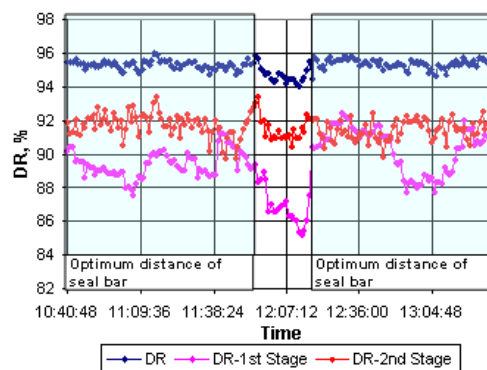
Optimum range Poor wash results





## Effect of seal bars distance on washing results

- The distance of the seal bar from the drum has fundamental effect on the DR-value
- An unbalanced seal bar caused wash liquid to escape from the 1st stage to the 2nd stage



## Conclusions of the PhD thesis

- The results shows that refractometer measurement works for enough accuracy in very demanding process conditions. By using refractometer it can be measured immediately and reliably liquid and pulp streams dissolved dry solids. It can be noticed that calibrations should be done for used wood species and for used installation place.
- For utilizing measuring results of three refractometer, consistency measuring and flow rate measuring at least it was possible to create the measuring system, and then calculate in real-time the effectiveness (DR or  $E_{10}$ ) of the single washer.
- The refractometers can be used for the searching of the washer's optimal operating point.

## Conclusions of the brown stock washing projects

- By controlling the performance of washers (cleanness of the screens, velocity of the screens, seal bars distance, rotation speed of drum etc.) and by changing process conditions (temperature, pH, consistency, wash water amount) it is possible to improve the effectiveness of washing line.
- Continuous measurements are needed for monitoring
- By using refractometers and data-analyses tools, it is possible to discover the black spots of the washing.
- This allows for improved washing efficiency and reduced water consumption, which had clear effect on the economy of the other sub process like evaporation, bleaching and waste water treatment. Better washing results can also improve the yield of fiberline (oxygen delignification and bleaching).
- Refractometers provide whole new possibilities for implementing higher level process optimization, e.g. at the whole washing department.

## Thanks



**Thank you for your attention!**  
**Questions?**