Model development for real oxygen delignification processes

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ABSTRACT: Previous extensive work has been done on modeling the oxygen delignification process, based on how the basic parameters, i.e., temperature, kappa number, concentration of alkali, and concentration of oxygen, affect the delignification rate. However, these models are not used extensively to evaluate the performance of real processes, primarily because they have not been able to properly consider all the essential issues affecting delignification in practice. Such issues include the mass transfer and consumption of oxygen, which defines the concentration of dissolved oxygen in the process, and the effect of that concentration on the delignification rate.

In this paper, a new way to model the oxygen delignification process is used in which these parameters, among other smaller matters, are taken into account. The basic model and its parameters were defined by the information obtained from the literature, delignification made in the laboratory tests, and mill processes and mill tests. An essential aspect of these studies was the information obtained from the oxygen concentration measured in the residual gas obtained from the top of the reactor. With the aid of this measurement, it was possible to define more accurately the consumption of oxygen and partial pressure of oxygen that define the concentration of dissolved oxygen in the reactor.

Using mill experiments, a model was formed that predicts the operation of the oxygen delignification process. The model was used to show how much the process could be improved by optimizing the charge of the oxygen. The mill experiments also confirmed that mass transfer of oxygen is modeled correctly enough, except when the charge of oxygen is very low and/or the mixing is not efficient enough. In that case, there is variation in the concentration of oxygen in the process that should be taken into account in the modeling.

Application: The described modeling approach can be useful in many ways in order to understand and improve oxygen delignification processes.

There have been many studies related to oxygen delignification and modeling the oxygen delignification process [1,2]. However, there have not been many real attempts to model the existing mill oxygen delignification processes. The main obstacle is that there has not been sufficient information related to the oxygen mass transfer that determines the concentration of dissolved oxygen, which in turn determines to quite an extent the speed of delignification. Recent studies [3-18] related to oxygen bubble size in delignification processes, the effect of bubble size on delignification, and the relationship between oxygen bubble size and mass transfer provide the possibility to model real oxygen delignification processes more accurately. There are also other factors in the processes that must be considered in the modeling, e.g., the effect of carryover and the use of white liquor as an alkali source.

This paper uses a semi-mechanistic model for the numerical evaluation of the real oxygen delignification process. The main purpose of this modeling is to use it as a tool that turns the results from the mill or laboratory tests into basic parameters that determine the delignification rate. In this way, modeling can be used to:

- Understand, develop, optimize, compare and even control existing processes.
- Understand the physics and chemistry of oxygen delignification better.

The main aims of the studies described in this paper were to:

- Improve the modeling by verifying those parameters that are related to oxygen.
- Develop and use new continuous measurements related to the oxygen delignification process, mainly image-based bubble measurement, measurement of the residual gas oxygen concentration, and measurement of the gas void fraction from the pulp.
- Improve the operation of the oxygen delignification process where these studies were conducted.

MODELING EQUATIONS AND ASSUMPTIONS

The modeling in this paper is mainly based on previous modeling done by van Heiningen et al. [2] and Käyhkö et al. [18]. The delignification rate is calculated as:

$$\Delta K = -A(3*10^6/60)e^{-51000/(8,314T)}([OH-])^{0.7}(CO_2)^{0.7}(K)^2\Delta t \quad (1)$$

where:

K: Kappa number
T: Temperature, K
[OH-]: Hydroxide ion concentration, mol/L
C_{o2}: Concentration of dissolved oxygen, mol/L
Δt: Value of 4 s was used in the numerical calculation
A: Constant that is set so that modeling fits the results.

We could have used other equations for the delignification rate such as that used by Yin et. al [1], for example, but since there was no information on which equation best suits the real processes, we decided to keep this approach. Further, this equation nicely explains that there are essentially four factors that should determine the delignification rate, which are described in the following sections.

Kappa number

In the calculation, a hexenuronic acid (HexA) free kappa number is used, since oxygen delignification should not affect the HexA content.

Temperature

With softwood especially, the temperature in the reactor may rise significantly, but in these calculations, a constant average temperature has been used for the reactor.

Hydroxide ion concentration

Hydroxide ions come into the process not only with added alkali, but also with carryover. The concentration in the beginning of process is obtained by summing these together. If white liquor is used as an alkali source, the OH- content was assumed to be 56% from the OH- content of the caustic soda. The [OH^-] ion consumption rate was calculated as:

$$\Delta[OH^-] = \frac{1.5 b_2}{40}\left(\frac{c}{100}\right)\Delta K \quad (2)$$

where:

c is pulp consistency, and b_2 is the stoichiometric coefficient (grams sodium hydroxide, NaOH, consumed/grams lignin removed); according to Violette [19], a value of 0.90 is used.

The value of 1.5 in Eq. 5 derives from the experience that 1 softwood kappa unit represents 1.5 g of lignin in 1.0 kg of pulp. The number 40 represents the molar mass of sodium hydroxide.

Concentration of dissolved oxygen

The dissolution of oxygen is based on the following equation:

$$N = k_L a(C_{O^2}{}^* - C_{O^2})\Delta t \quad (3)$$

where:

N: Dissolution of oxygen, g/L

C_{O2}: Oxygen saturation constant, calculated here based onHenry's law, e.g., at temperature 95°C and pressure of 8.3 bar; the value is 0.2 g/L.

C_{O2}: Concentration of dissolved oxygen, g/L

The calculation of k_{La} is based on the basic film mass transfer equation:

$$k_L a = \frac{D_{O^2} A}{sV} \quad (4)$$

where:

k_{La} : Mass transfer rate, 1/s

D_{O2}: Diffusion coefficient of oxygen; the value used in these calculations was $5.7 * 10^{-9}$ m^2/s.

s: Distance over which oxygen is diffused

A: Surface area of the gas bubbles

V: Volume of the system

When the gas void fraction (Xg) is known, the equation can also be written as:

$$k_L a = \frac{12 D_{O^2} X_g}{B*((1/X_g)^{1/3}-1) {d_b}^2} \quad (5)$$

where:

X_g: Gas void fraction

d_b: Bubble diameter, m

B: Constant, 4.0

This equation has not been verified yet. In the delignification process, there are several existing phenomena that indicate this equation may give too-large k_{La} values, e.g., oxygen bubbles and fibers are not homogenously spread in the reactor. This is why that in the modeling, a constant was used that decreases the k_{La} value. The value used was 4.0.

The oxygen consumption rate r_{O2} (mol/L) is related to the rate of decrease of the kappa number:

$$r_{O^2} = \frac{1{,}5 b_1}{32}\left(\frac{c}{100}\right)\Delta K \quad (6)$$

where:

i_1 is the stoichiometric coefficient (grams oxygen, O_2) consumed/grams lignin removed); the number was set on the basis of the mill measurements.

A value of 1.5 in Eq. 5 derives from the experience that 1 softwood kappa unit represents 1.5 g of lignin in 1.0 kg of pulp. The number 32 represents the molar mass of the oxygen. If an oxidized white liquor was used, it was assumed that the oxidation state was 75% [20]. Hence, 1 kg of oxidized white liquor decreased the oxygen charge 0.15 kg.

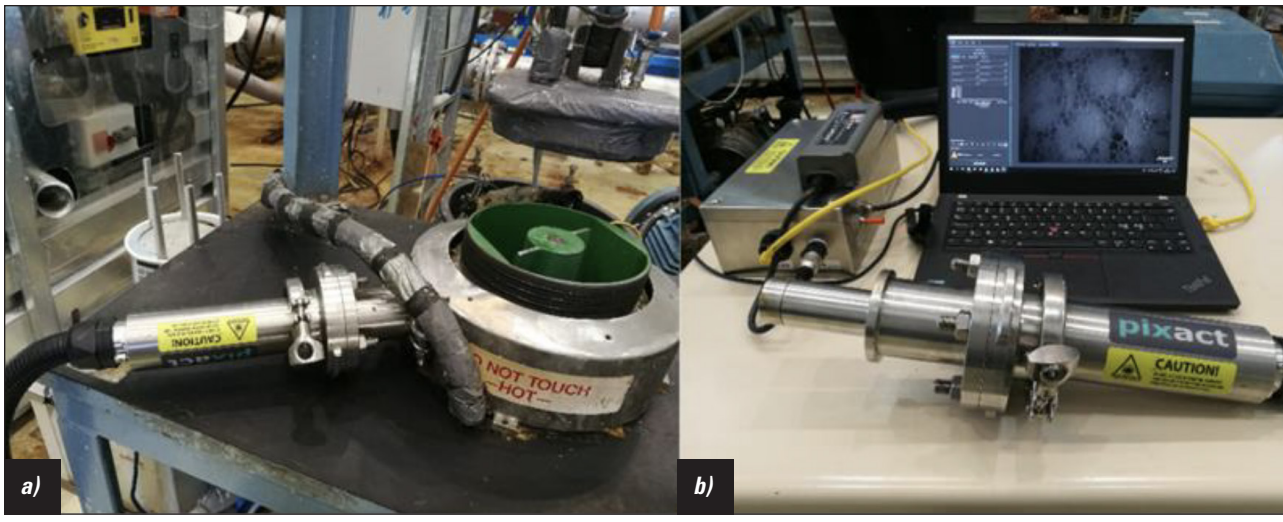
The saturation concentration of oxygen was determined according to Henry's law based on pressure and temperature. The pressure at the height of h in the reactor (m) is calculated as:

$$p = p_{in} - 0.1h \quad (7)$$

where p_{in} is pressure in the feed of the reactor (bar).

<u>The carryover</u>

The amount of lignin (or other substances that react with oxygen and consume alkali) in the water phase is assumed to be 1/3 x chemical oxygen demand (COD) value. It is also assumed that a certain proportion of this lignin is already oxidized (70%), and hence does not affect the process. The



1. (a) Mark reactor and (b) bubble measurement used in the laboratory delignification.

value obtained is changed to a kappa value, assuming that 1 kappa unit is the same as 1.5 g lignin/kg pulp. In the modeling, it is also assumed that dissolved lignin behaves similarly to lignin attached to fiber.

Also, when the pulp is not totally washed, as in the mill, lignin is diffusing from the fiber wall at a high temperature in a caustic environment [21], and also, addition of alkali causes alkaline leaching [22]. Delignification would happen in the oxygen delignification process, even if there was not any oxygen present. This leaching phenomenon is taken into account in the modeling by assuming that lignin will dissolve 2 kappa units at the beginning of the process.

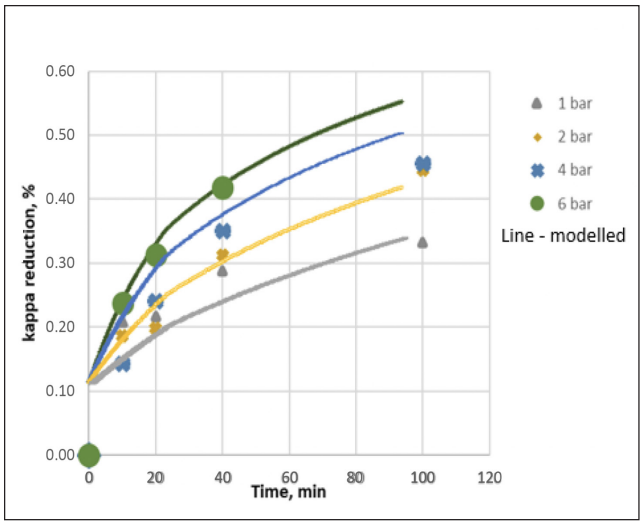
EXPERIMENTAL

The results were obtained by collecting information from softwood mill oxygen delignification processes and undertaking delignification with a modified Mark reactor (**Fig. 1**), including bubble size measurement, pressure and temperature recording, automatic temperature control, and gas flow meter and valve for the sampling. The bubble size measurement (Fig. 1) was described in an earlier publication [5].

The pulp used in the laboratory delignification was taken from the mill from the feed of the oxygen delignification stage. Its kappa number was 26.5, consistency 12%, and dissolved COD 20 g/L. Temperature in the delignifica-

Kappa in	28.5	OWL charge I, kg NaOH/o.d. metric ton	20.4
HexA (assumed)	2	Carryover NaOH, kg/o.d. metric ton (assumed)	14
Kappa after reactor I	18	Total NaOH I, kg/o.d. metric ton	34.4
Kappa after reactor II	13.7	OWL charge II, kg NaOH/o.d. metric ton	13
Alkaline leaching	2	Carryover COD, kg/o.d. metric ton	300
Pressure I (feed), bar	7.3	Dissolved lignin, kg/o.d. metric ton	100
Pressure I (top), bar	3.4	Dissolved kappa	62.5
Pressure II (feed), bar	9.2	Dissolved unoxidized kappa	12.4
Pressure II (top), bar	3.3	A, Kappa reduction fix reactor I	4
Average temperature I, °C	96	A, Kappa reduction fix reactor II	0.8
Average temperature II, °C	100	b1, O_2 consumed in reactor I	0.36
Oxygen charge I, kg/o.d. metric ton	11.3	b1, O_2 consumed in reactor II	0.4
Oxygen charge II, kg/ o.d. metric ton	6	b2,OH- consumed	0.9
Oxygen purity, %	93	Retention time reactor I, min	31
Pulp consistency, %	12	Retention time reactor II, min	141
HexA: hexanuronic acid; OWL: oxidized white liquor; NaOH: sodium hydroxide; COD: chemical oxygen demand; O_2: oxygen; OH-: hydroxide ion.			

I. Values used in the modeling.



2. Effect of oxygen pressure on the delignification obtained in laboratory tests and according to modeling.

tion was 90°C, pulp consistency was 10%, NaOH charge was 30 kg/metric ton, magnesium hydroxide (MgOH) charge was 0.5 kg/metric ton, and the gas void fraction (*Xg*) was 10%. The pulp was mixed at 2400 rpm for 10 s after the Mark reactor was pressurized with oxygen and before every sampling, and the pressure was kept constant during the experiment. The core idea for this setup was to keep the amount of dissolved oxygen at the saturation level throughout the experiment. According to the bubble size measurements, the oxygen bubble size in the experiments was normally below 50 µm, which should ensure this.

The kappa measurement was done according to standard ISO 302:2015 "Pulps — Determination of kappa number." The continuous gas void fraction (GWF) measurement from the DD-washer feed pulp was done using a clamp-on Ackumen Echowise gas analyzer (Buckman; Memphis, TN, USA). The oxygen concentration measurement was done using a Geotech G110 (Geotech; Denver, CO, USA) gas analyzer.

MODELING BASED ON LABORATORY AND MILL RESULTS

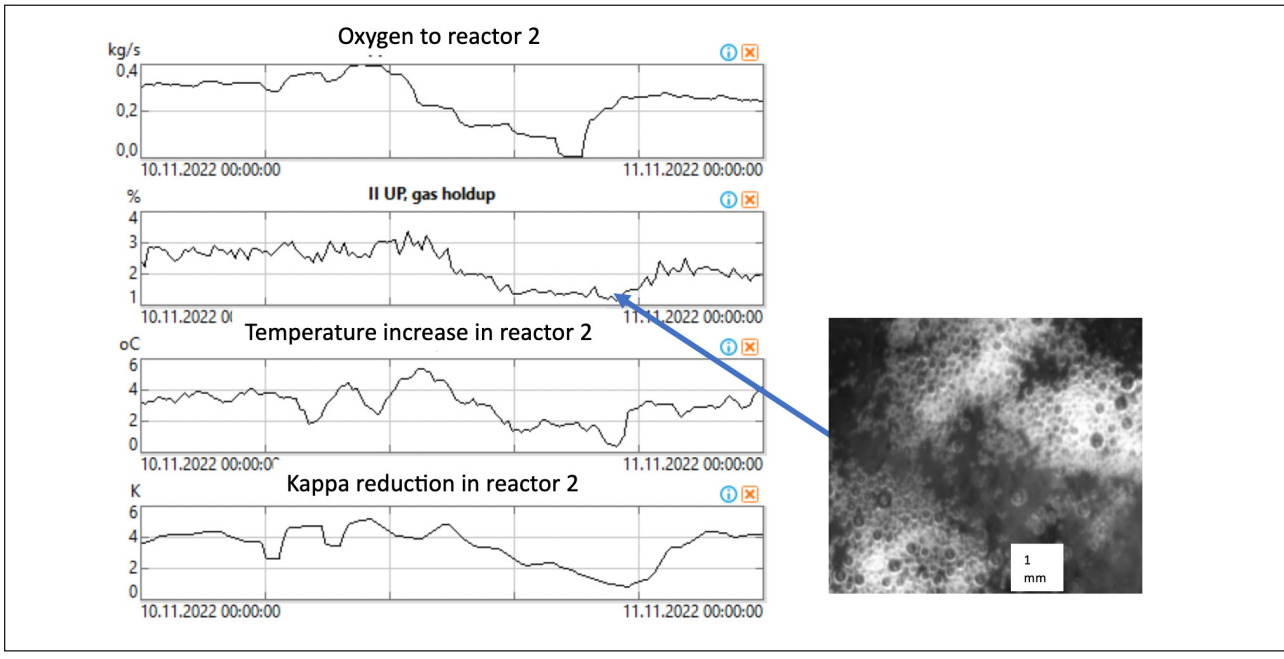
The main target in this study was to verify the parameters related to oxygen in the modeling. Those parameters are:

- The effect of the concentration of dissolved oxygen on the delignification rate in Eq. 1.
- The parameters that define the concentration of dissolved oxygen, mainly Eq. 5, which defines the mass transfer of oxygen from the gas phase to liquid and reaction sites and the consumption of oxygen in the process, as in Eq. 6.

The values used in the modeling are shown in the Experimental section and otherwise are the same as in the mill modeling (**Table I**), except that the first kappa reduction fixing value was 2 instead of 4, which was obtained in the mill modeling. This indicates that delignification in the mill occurs faster than in the laboratory, but this result may be ignored; e.g., the pulp sample used in the laboratory was not taken at the same time as the mill tests were made.

The effect of oxygen pressure on the delignification is shown in the **Fig. 2**. There is some deviation in the results, but the modeling seems to predict quite well the effect of oxygen pressure, i.e., the concentration of dissolved oxygen on the delignification.

Mill studies were conducted at the Finnish softwood pulp mill. Earlier, it was observed [18] that in this two-stage



3. Effects of the oxygen charge in reactor 2.

4. The oxygen content of the residual gas in the top of reactor 2 was nearly zero.

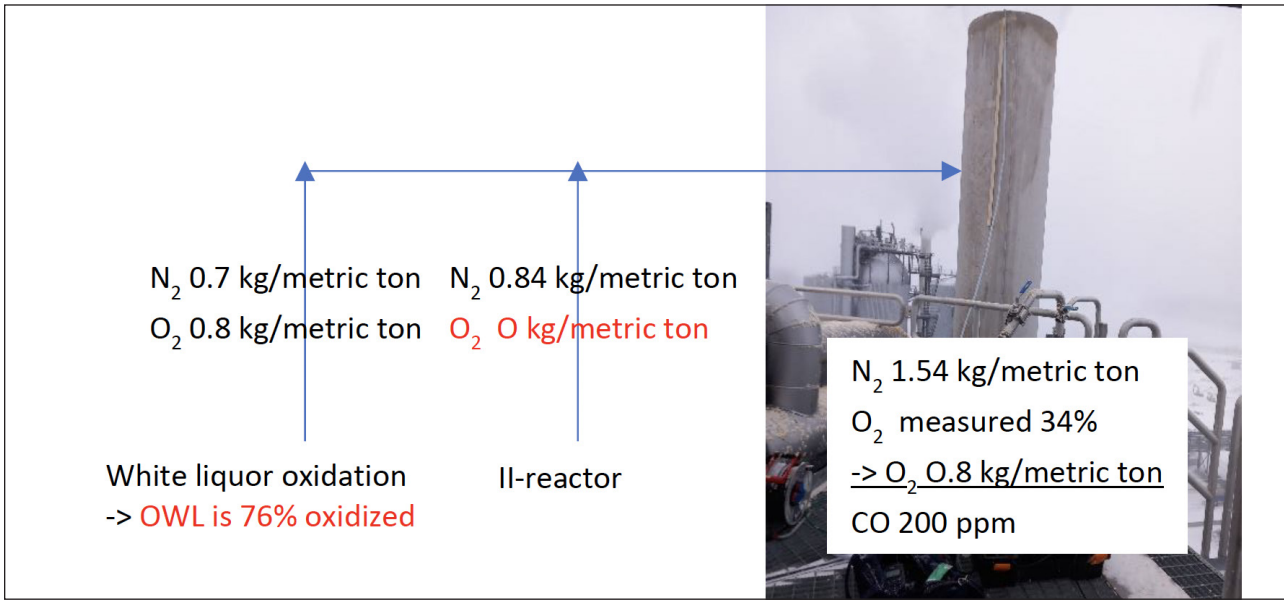
oxygen delignification process, the average bubble size of the oxygen in the feed of the reactors was very small, about 0.1 mm. In addition, there was clearly a large amount of residual gas in the top of the reactors, and hence the modeling indicated that both reactors were practically saturated with dissolved oxygen. If this is true, then the charge of the oxygen could be decreased without decreasing the kappa reduction. This was not the case in the mill tests, as seen in **Fig. 3**. When the oxygen charge was decreased in the second reactor, the kappa reduction also decreased, and there was still a large amount of residual gas in the top of the reactor. This was a very odd result. There was a possibility to take filtrate from the pulp in the top of the reactor, get the gas sample, and measure its oxygen content, and it was nearly zero, as seen in **Fig. 4**.

As it turned out, this mill was not using liquid oxygen. They have an oxygen-producing plant, and the purity of the oxygen is 93 ± 0.5%, so the nitrogen content of the gas is 7%. This does not sound like much, but it has a clear effect on the process in this case. In addition, using bubble measurement to measure the amount of residual oxygen is not working, since there is always residual gas in the top of the reactors with a varying concentration of oxygen.

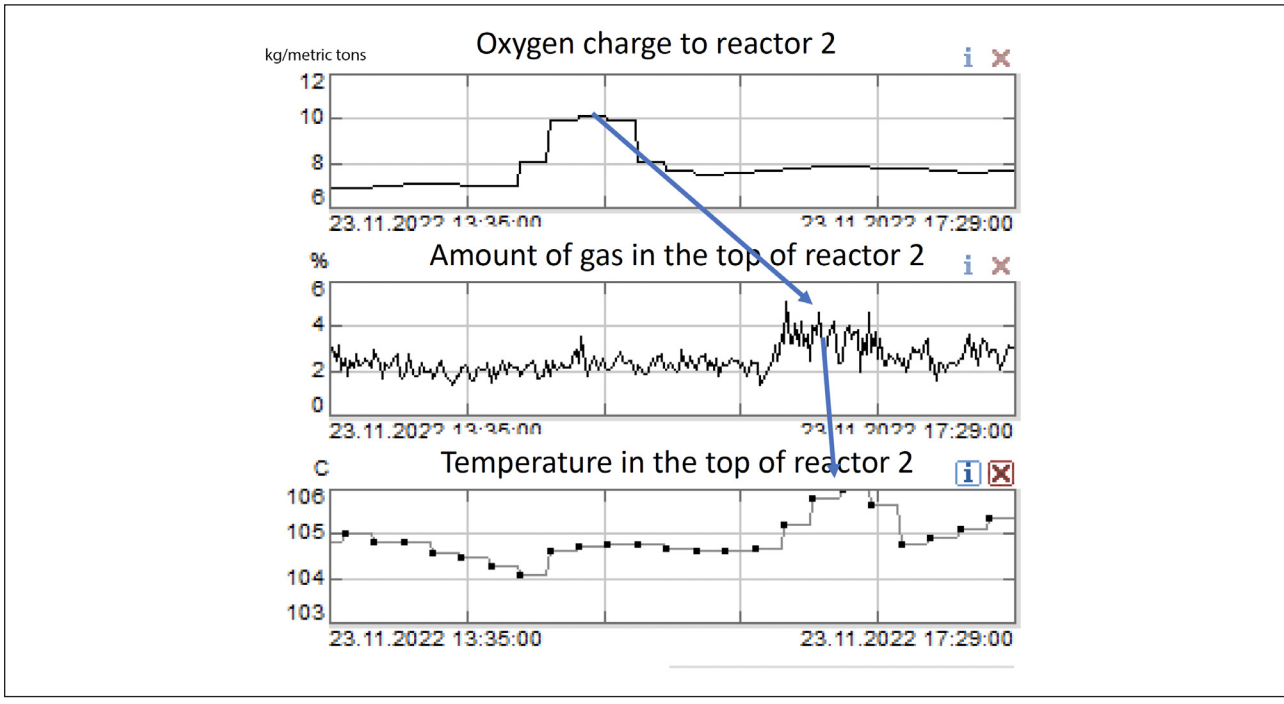
However, the existence of a known amount of nitrogen in the process also gives the possibility to quantify the amount of residual oxygen (kg/metric ton) in the process by measuring the oxygen concentration from the purge gas. In this mill, it was possible to obtain a proper gas sample from the purge stream where residual gases from the white liquor oxidation and second O_2 reactor are connected, as shown in **Fig. 5**. According to mass balance, the concentration of oxygen in the second reactor residual gas was 0 kg/metric ton. Here, information could also be obtained on how far the white liquor is oxidized. This measurement indicates that it is 76% oxidized. According to [5], the typical value is 75%, and that value was also used in the modeling.

If the concentration of oxygen in the top of the reactor is low, increasing the charge of the oxygen should speed up reactions, increase the consumption of oxygen, and hence, also increase the temperature in the top of the reactor. In the test undertaken in the second reactor, this was the case, as shown in **Fig. 6**. When the oxygen charge was increased by 3 kg/metric ton, the temperature in the top of the reactor increased about 1°C. The amount of residual gas in the top of the reactor also increased, but only a little, which indicates that any additional oxygen was mainly consumed.

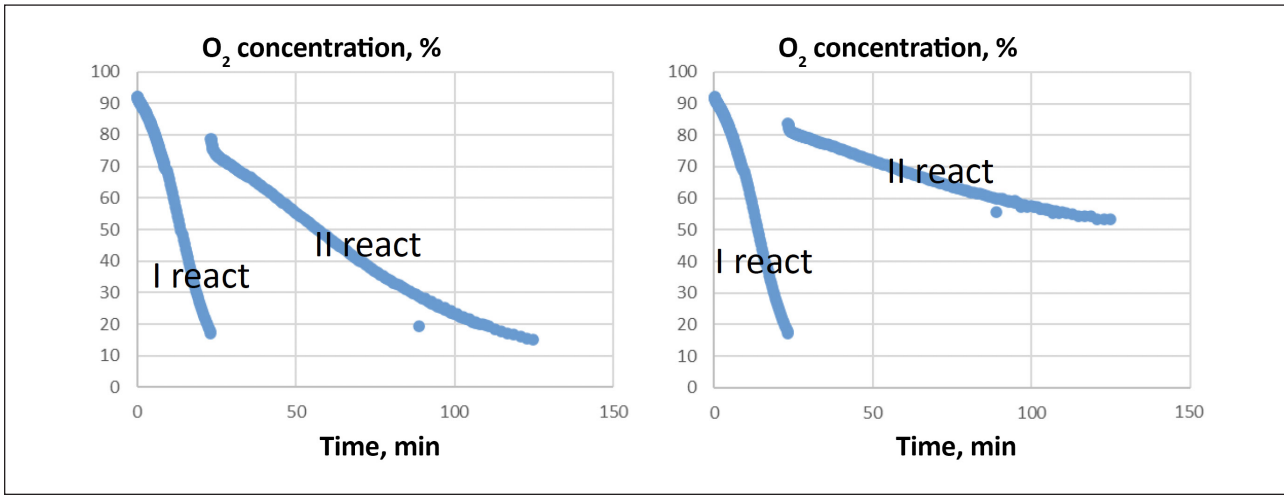
According to the oxygen delignification modeling, the following result for the concentration of dissolved oxygen in the reactors is obtained, as shown in **Fig. 7**. When the



5. The mass balance of nitrogen (N_2) and oxygen (O_2) in the system (OWL: oxidized white liquor; CO: carbon monoxide).



6. Effect of oxygen charge on the amount of residual gas and temperature in the top of the second reactor.



7. (a) Concentration of oxygen in the reactors without oxygen charge increase and (b) with oxygen charge increase of 3 kg/metric ton in reactor 2.

oxygen charge to reactor 2 is increased by 3 kg/metric ton, the concentration of dissolved oxygen in the top of reactor 2 is multiplied. According to the modeling, this should increase delignification by 0.9 kappa units and increase the temperature by 1.0°C.

According to these results, it would be beneficial in this process to increase the charge of oxygen. Residual gases cause some vibrating problems in reactor 1, and the residual gases from reactor 2 cause problems in the subsequent washing stages. Because of these problems, the mill wants to keep the charge of oxygen as low as possible.

Lately, the mill has made investments that make it possible to remove residual gases from the pulp in the top of the reactors. At the same time, sample points were added to both reactors to obtain samples from the residual gas, as shown in **Fig. 8**. This offers the possibility to gain new essential information from the process to be used in the modeling. Oxygen concentration directly tells the partial pressure of oxygen in the top of the reactor, which then defines the amount of dissolved oxygen. In addition, since the amount of nitrogen fed into the process is known, the amount of residual oxygen (kg/metric ton) can be calculated, and the exact amount of oxygen consumed in the reactor is then also obtained.

Oxygen charge to reactor 1 was increased from 11 to 14 kg/metric ton. At the same time, the concentration of

8. Sampling point and measurement for the residual gas in reactor 2.

oxygen was measured from the top of the reactor, and it increased from 15% to 55%, as shown in **Fig. 9**. Simultaneously, the temperature in the top of the reactor increased by about 0.7°C, and the kappa decreased about 1.3 kappa units.

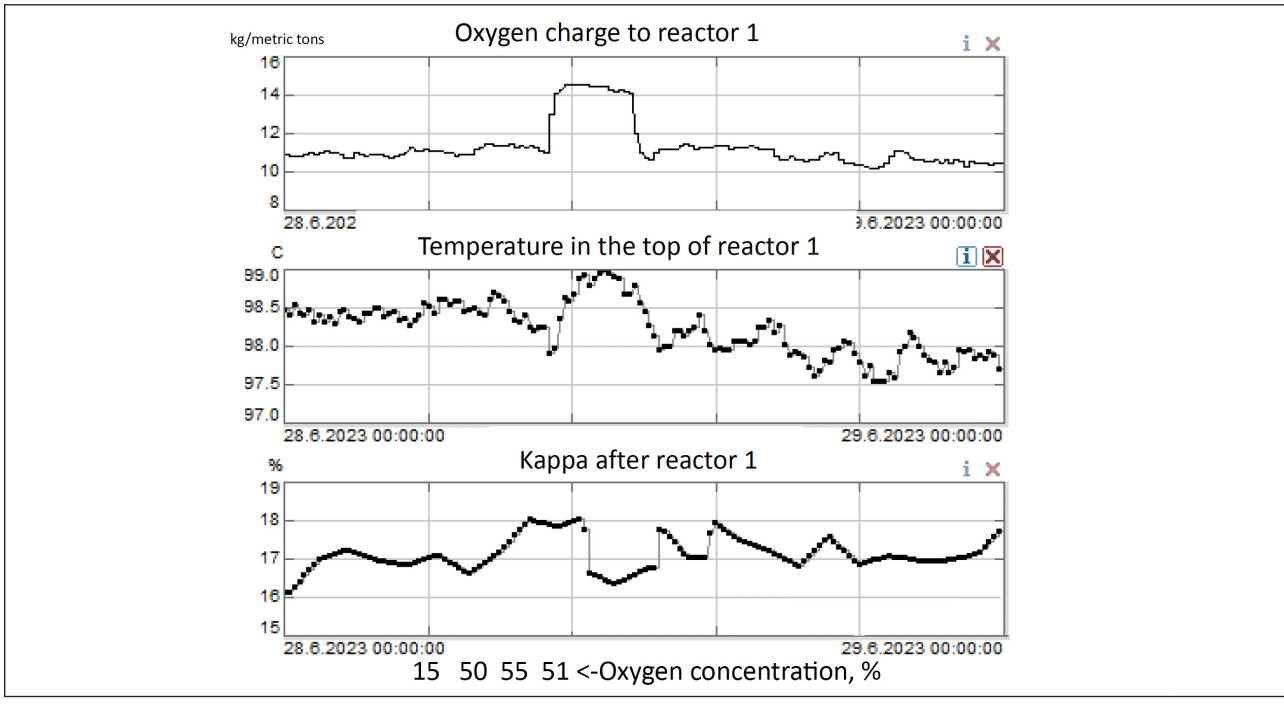
The model was fixed so that concentration of oxygen in the residual gas was set to 15% in the top of the reactor 1, and the kappa reduction after both reactors was set to same in the model as observed in the mill, as shown in **Fig. 10**. Values used in the modeling are shown in the **Table I**.

According to the modeling, a 3 kg/metric ton charge increase to the first reactor should decrease the kappa after the first reactor by 2.0 kappa units, which is a little higher than that observed in the process (Fig. 9). In addition, the oxygen concentration in the top of reactor 1 should rise to 55%, which was the same as that measured in the mill.

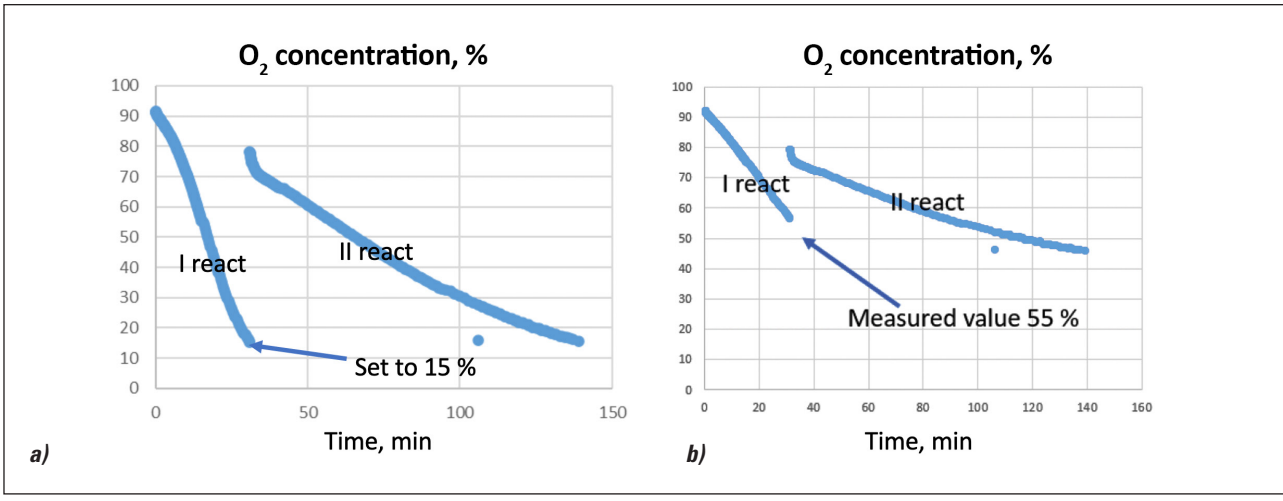
In another test charge increase, the temperature increase and kappa decrease were almost the same, but the oxygen concentration in the top of the reactor increased only to 25%, as shown in **Fig. 11**. In addition, according to the modeling, it should have risen to 47% (**Fig. 12**).

In the test undertaken in reactor 2, a similar result was obtained (**Fig. 13**). The charge of oxygen was raised 3 kg/metric ton and the temperature increased and the kappa decreased, but the concentration of oxygen did not increase at all. On the other hand, the result is understandable; oxygen is consumed more and kappa is decreased, but this is not at all suitable for modeling. If the concentration of oxygen is not increased in the gas phase in the top of the reactor, then, according to the modeling, the concentration of dissolved oxygen is not increased, hence there is no driving force for delignification reactions and a decrease in the kappa should not happen.

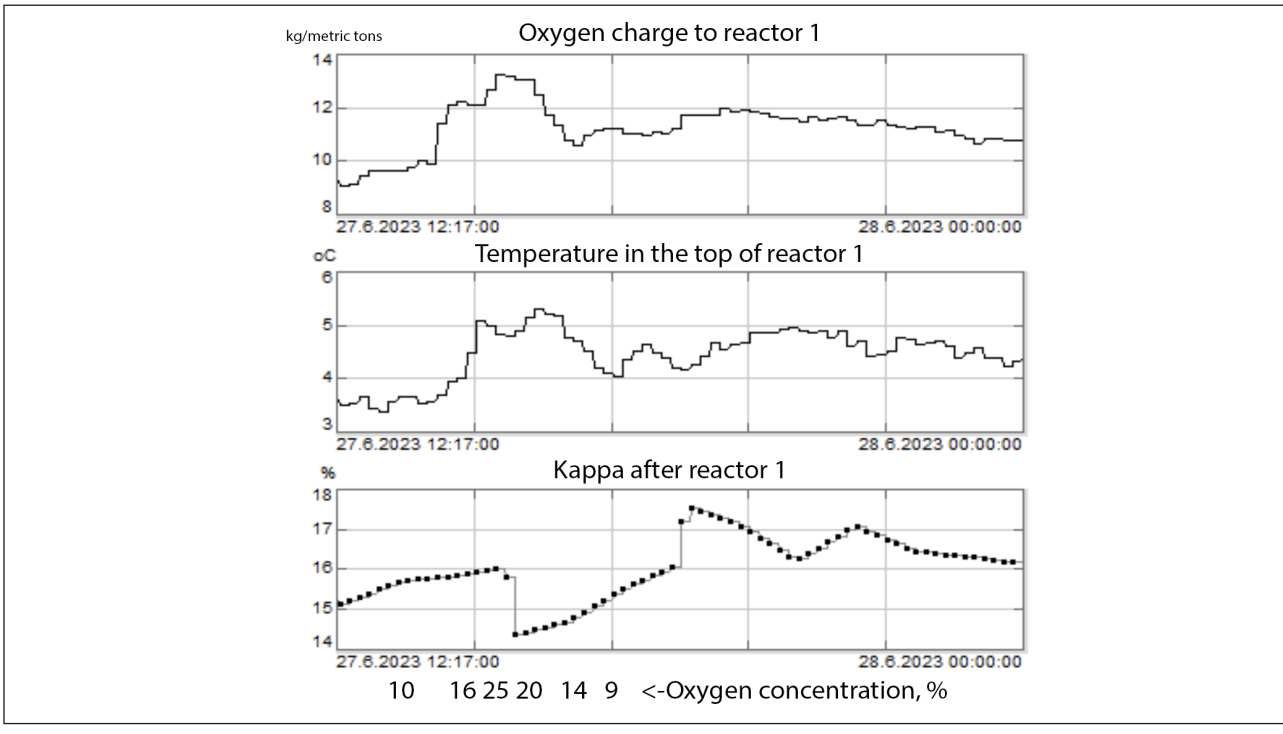
One explanation for this is that the distribution of oxygen gas in the reactor is not even and there are places where the concentration of oxygen is very small or even zero. This seems to be the case, especially in reactor 2. The



9. Oxygen charge to reactor, kappa after reactor, oxygen concentration, and temperature in the top of the reactor during the mill trial.



10. (a) Concentration of oxygen (O_2) in the reactors without oxygen charge increase and (b) with oxygen charge increase to reactor I by 3 kg/metric ton.



11. Oxygen charge to the reactor, kappa after reactor, oxygen concentration, and temperature in the top of the reactor during the mill trial.

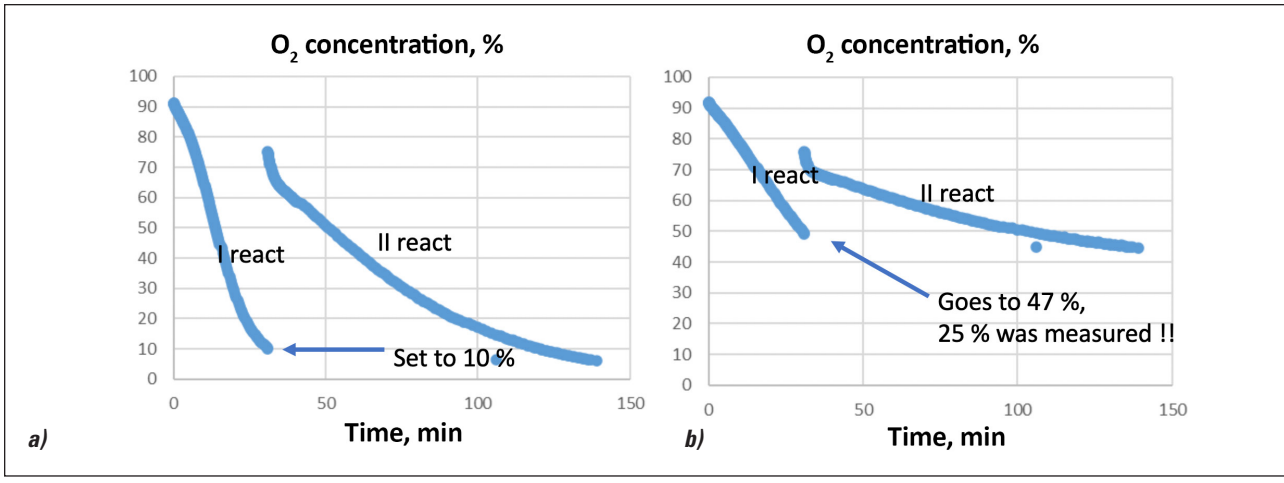
motor in the oxygen mixer in reactor 2 is much smaller compared to reactor 1, which may partly explain this difference. A more probable explanation is related to feeding of oxygen. This mill is using much less oxygen than what was originally planned when the mill was built. The diameter of the feed pipe, which is located under the pulp feed pipe, is big, and hence the speed of the oxygen gas, especially in the reactor 2, is very small. In that case, pulp may enter into the oxygen feed pipe and cause plugging and variation to the charge of oxygen, which the mixer cannot even out.

Effect of residual gases on washing

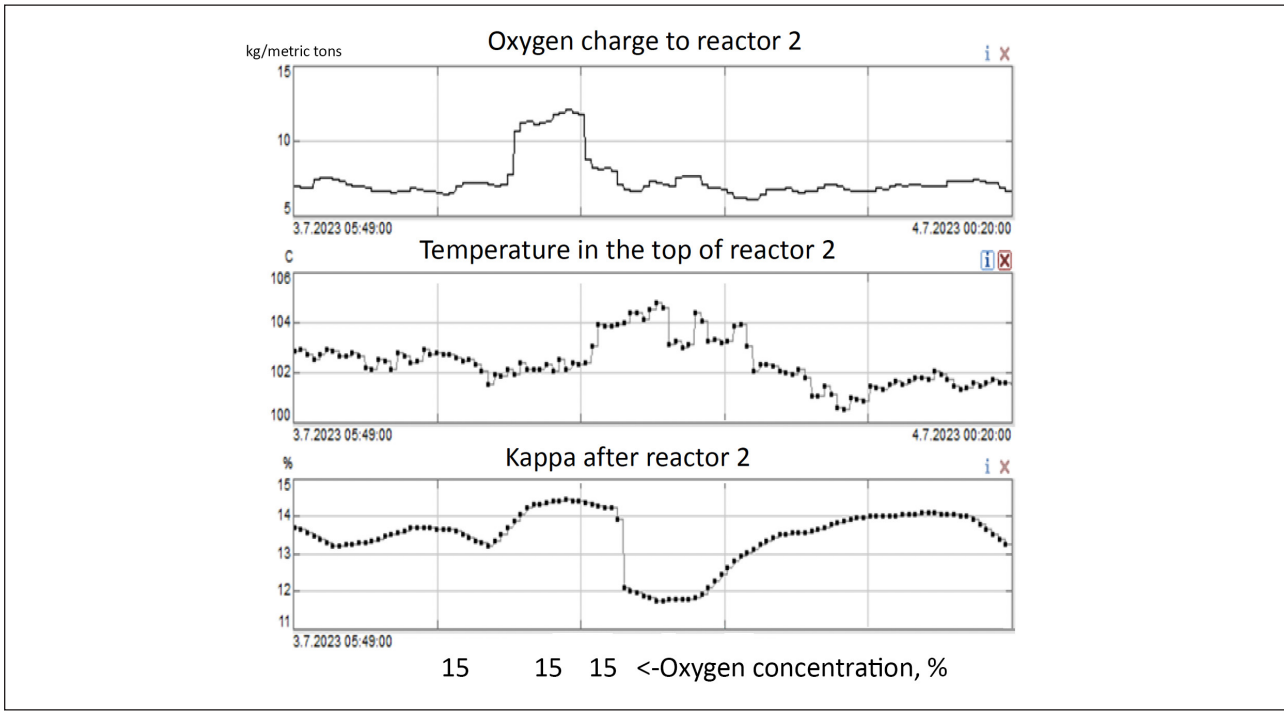
Residual gases may have a negative effect on the performance of the post-oxygen washing. Two gas measurements have been installed in the feed of the DD-washer after oxygen delignification. **Figure 14** shows that there is a large amount of variation and the gas content can also be quite high.

Figure 15 shows the gas content based on the Echowise and bubble imaging-based measurement, as well as an example of the bubble image. These also indicate that gas content in the feed of the DD-washer can be quite high.

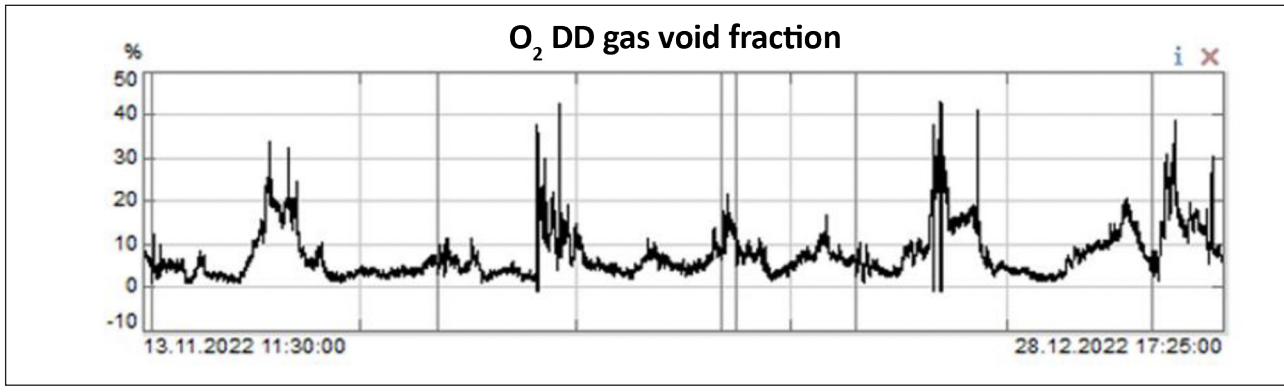
During the mill test, the aim was also to gain information



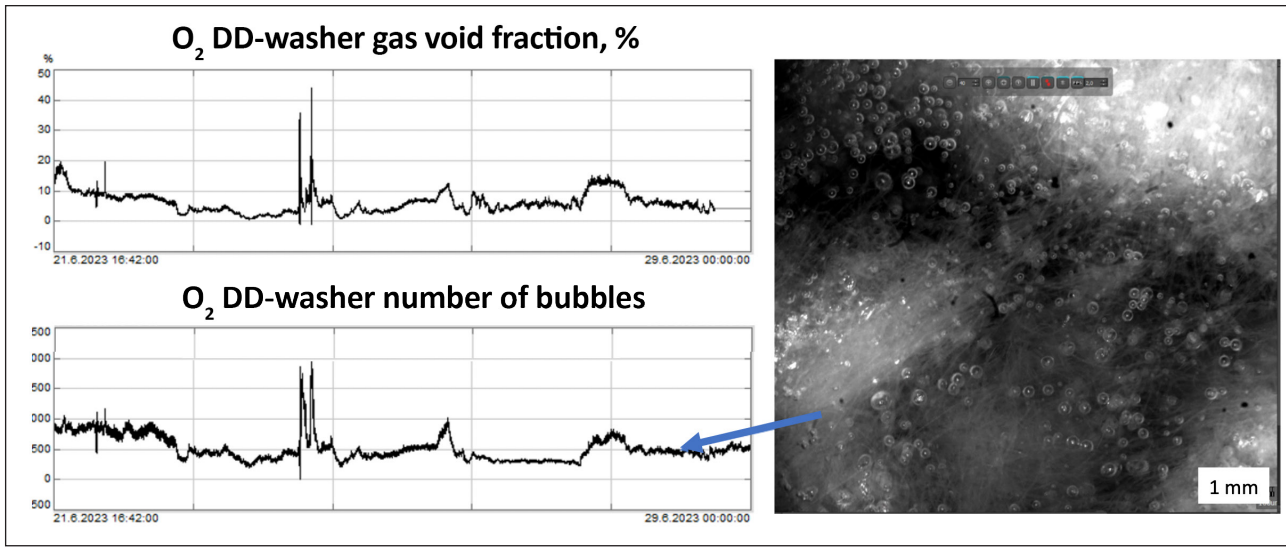
12. (a) Concentration of oxygen in the reactors without oxygen charge increase and (b) with oxygen charge increase by 3 kg/metric ton.



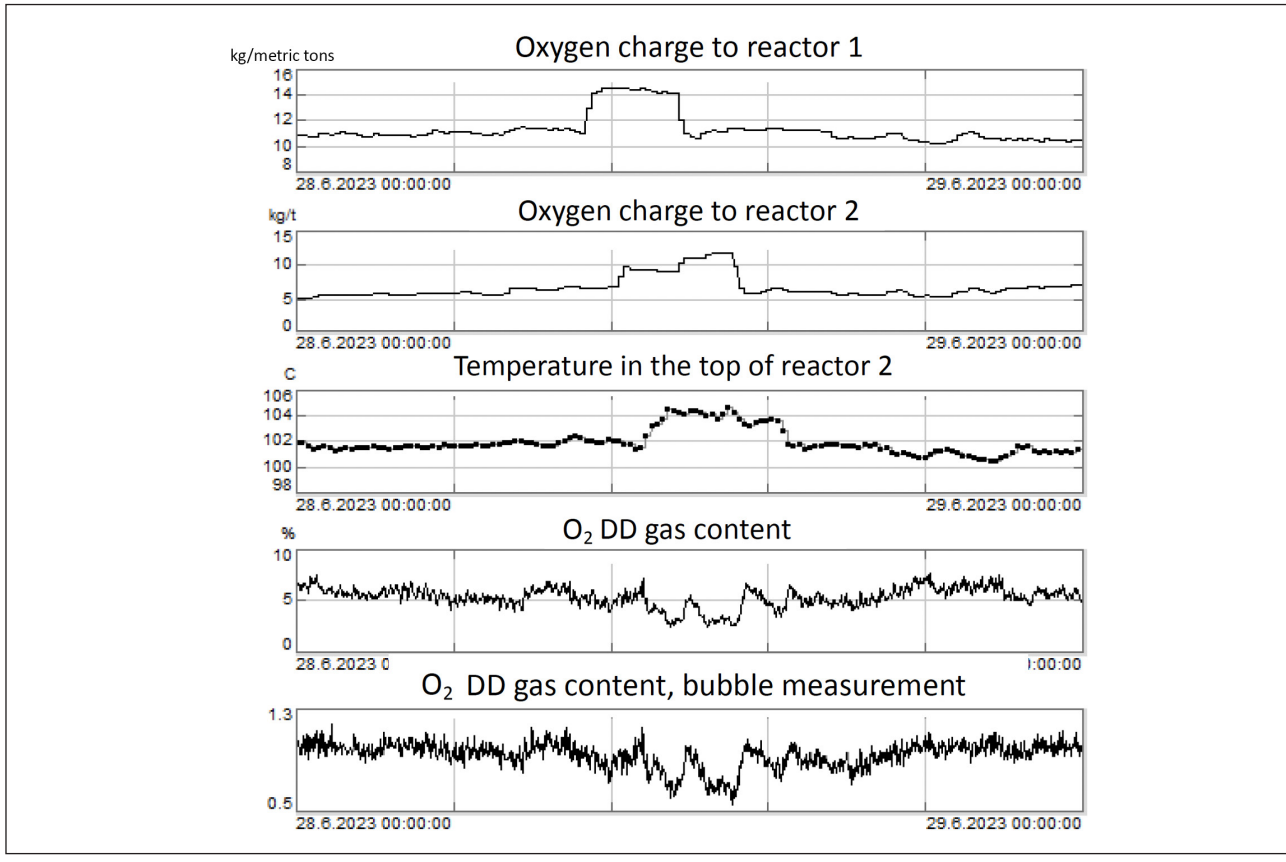
13. Oxygen charge to reactor, kappa after reactor, oxygen concentration and temperature in the top of the reactor during the mill trial.



14. Gas content in the feed of the DD-washer measured with Echowise gas measurement.



15. Gas content in the DD-washer feed pulp based on the Echowise and bubble imaging-based measurements, along with an example of the bubble image.



16. Effect of oxygen charge on the gas content in the feed of the DD-washer pulp and the temperature in the top of the second reactor.

on how increasing the oxygen charge increases the gas content in the feed of the DD-washer (**Fig. 16**). This information was not obtained, because the oxygen charge increased the temperature in the top of reactor 2; this increased vaporization of water, and hence removal of gases from pulp decreased the gas content in the washer.

Future investigations will consider if the new gas removal systems in the top of the reactors are working well enough that oxygen charges could be increased and perhaps well enough that removal of residual gases by flashing is no longer necessary so that process temperatures could be decreased.

CONCLUSION

In this paper, the modeling of oxygen delignification processes was developed further and used to analyze mill results. The modeling seems to work well, fit the laboratory and mill results, and give some indications about the operation of existing oxygen delignification processes, including:

- The concentration of residual gas was taken as a new measurement in modeling the real oxygen delignification processes. On the basis of this measurement, the oxygen gas partial pressure and the consumption of oxygen, and hence the concentration of dissolved oxygen in the reactor, could be modeled more precisely. Measurement of the concentration of the oxygen in the residual gas may give a great deal of valuable information for development of the modeling, especially when the development is based on the mill data.
- Continuous residual gas oxygen concentration measurement could be used to optimize the feeding of oxygen in practice. This kind of measurement is currently under development and testing. In addition, continuous gas content measurements could be used to control gas-based problems in the washing.
- The results indicate that modeling oxygen mass transfer may work well enough. In addition, in some cases, especially when the charge of oxygen is low and when the mixing of oxygen is not very strong, the concentration of oxygen in the reactor may vary so much that this has a negative effect on the delignification, and the modeling does not work as such.

There are still many open questions related to modeling. However, modeling provides a base to extract essential information from laboratory tests and mill processes. Therefore, it can also be used to analyze and predict the operation of mill processes and to further develop the modeling itself. **TJ**

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ABOUT THE AUTHORS

At FiberLaboratory, we have conducted research work related to the mixing phenomena in pulp and paper processes for over 20 years. Mixing of gases has been one subject, and there have been indications that in the oxygen delignification process in particular, there is potential to improve the process by better understanding the state and behavior of oxygen gas dispersion. Previously, we and others have done lots of work related to oxygen delignification and modeling of the process, and this is the first time that modeling has really been used and developed for an actual process.

The most difficult aspect of this study was that making structural changes in the process takes a great deal of effort and time. This is something one must prepare for at the outset of such a study.

Through this study, we obtained information for development of the modeling, as well as experience on how to use modeling in mill studies in order to predict the effect of different changes in the process. Our most surprising finding was that modeling really helped by providing evidence that there can be, in this case, problems in the feeding of the oxygen, as was expected. We gained experience in how to use modeling in mill studies in order to predict the effect of different changes in a process.

In the short term, modeling and new measurements may help mills to optimize their oxygen delignification process. In the long term, this can yield improvements related to structure, control, and chemistry of the process.

Our next step is to perform more studies, especially at different mills, and at the same time, develop new continuous in-line measurement technologies and modeling.

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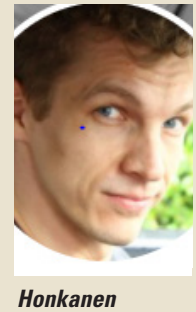
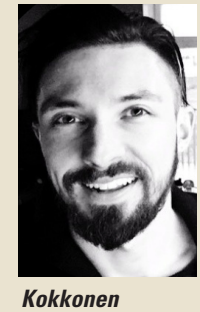

Honkanen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