

Model development for real oxygen delignification processes

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Summary

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, delignifications 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 which define the concentration of dissolved oxygen in the reactor. Using mill experiments, a model was formed which 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, which should be taken into account in the modeling.

Introduction

There have been many studies related to oxygen delignification and modeling the oxygen delignification process [1,2,**Error! Reference source not found.**]. However, up until now, 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

[3,5,6,7,8,9,10,11,12,13,14,15,16,17,25] 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 which must be considered in the modeling, e.g. the effect of carryover and the usage 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 which 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 which 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. [25]. The delignification rate is calculated as:

$$\Delta K = -A(3 \cdot 10^6 / 60) e^{-51000 / (8,314T)} ([OH^-])^{0.7} (C_{O_2})^{0.7} (K)^2 \Delta t \quad (1)$$

where:

K: Kappa number

T: Temperature, K

[OH⁻]: Hydroxide ion concentration, mol/l

C_{O₂}: Concentration of dissolved oxygen, mol/l.

A: a constant which is set so that the modeling fits the experimental results. This modeling uses a Hexa free kappa number and thus demands some correction especially in the case of the hardwood process.

We could have used other equations for the delignification rate, for example that used by Yin et. al [1], 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 which should determine the delignification rate:

Kappa number:

In the calculation, a 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;

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

where;

c: pulp consistency

b₂: stoichiometric coefficient (g NaOH consumed / g lignin removed), according to Violette [18] a value of 0.90 is used.

The value of 1.5 in equation (5) derives from the experience that one softwood kappa unit represents 1.5 grams of lignin in 1.0 kilogram of pulp. The number 40 represents the molar mass of sodium hydroxide.

Concentration of dissolved oxygen:

The dissolution of oxygen is based on the 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 in here based on Henry's law e.g., in 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 m²/s

s: Distance over which oxygen is diffused, see figure 1 below

A: Surface area of the gas bubbles

V: Volume of the system

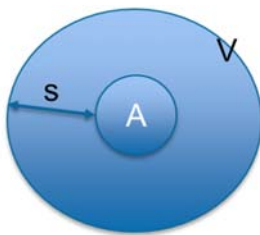


Figure 1. Basis for the calculation of $k_L a$.

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

$$k_L a = \frac{12D_{O_2} X_g}{B * ((1/X_g)^{1/3} - 1) d_b^2} \quad (5)$$

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 which indicate that this equation may give too-large KLa values e.g., oxygen bubbles and fibers are not homogenously spread in the reactor. That is why in the modeling a constant was used which decreases the kLa value. The value used was 4.0.

The oxygen consumption rate r_{O_2} [mol/l] is related to the rate of decrease of the kappa number as;

$$r_{O_2} = \frac{1,5b_1}{32} \left(\frac{c}{100} \right) \Delta K \quad (6)$$

where;

b_1 : Stoichiometric coefficient (g O_2 consumed / g lignin removed): the number was set on the basis of the mill measurements.

A value of 1.5 in equation (5) derives from the experience that one softwood kappa unit represents 1.5 grams of lignin in 1.0 kilogram 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 % [18]. Hence one kilogram 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} : Pressure in the feed of the reactor, bar

The carryover: The amount of lignin (or other substances that react with oxygen and consume alkali) in the water phase is assumed to be $1/3 * COD$ value; it is also assumed that a certain proportion of this lignin is already oxidized (70 %) and hence does not affect the process. The value obtained is changed to a kappa value, assuming that one kappa unit is the same as 1.5g 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 [26] and delignification would occur in the oxygen delignification process even without any oxygen present. This leaching phenomenon is taken into account in the modeling by assuming that lignin will dissolve three 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. Temperature in the delignification was 90 °C, pulp consistency was 10 %, NaOH charge was 30 kg/t, MgOH charge 0,5 kg/t and the gas void fraction (X_g) 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.



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

The kappa measurement was done according to standard ISO 302:2015. The continuous gas void fraction (GWF) measurement from the DD-washer feed pulp was done using a clamp-on Echowise gas analyzer. The oxygen concentration measurement was done using a Geotech 110 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 equation (1)

- The parameters which define the concentration of dissolved oxygen, mainly equation (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, equation (6)

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

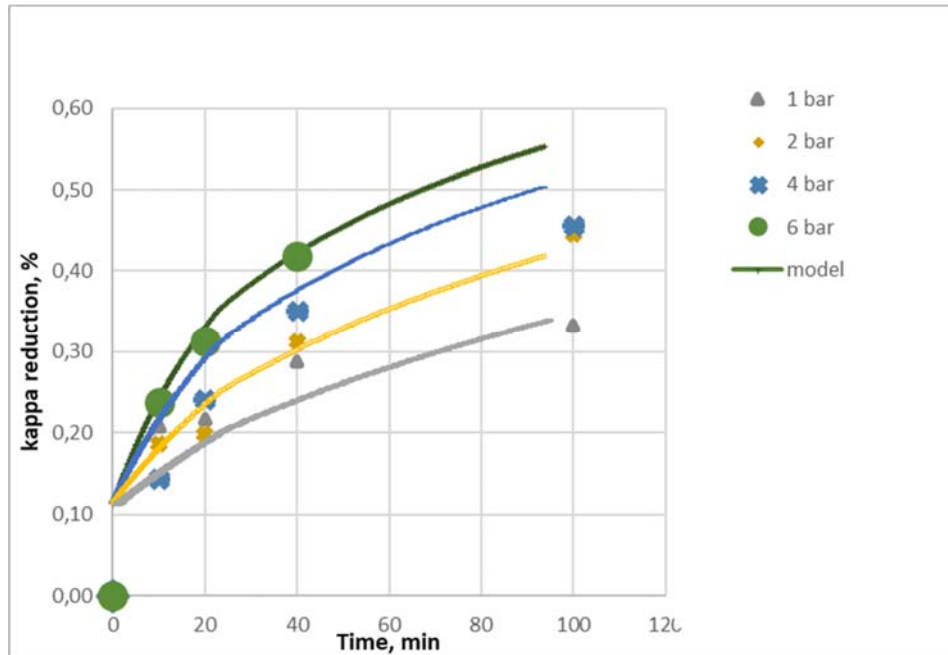


Fig. 2. The effect of oxygen pressure on the delignification obtained in laboratory tests and according to modelling.

Mill studies were conducted at the Finnis softwood pulp mill. Earlier it was observed [25] that in this two-stage 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 (see Fig. 3). When the oxygen charge was decreased in the first reactor, the kappa reduction also decreased and there was still a large amount of residual gas in the top of the reactor.

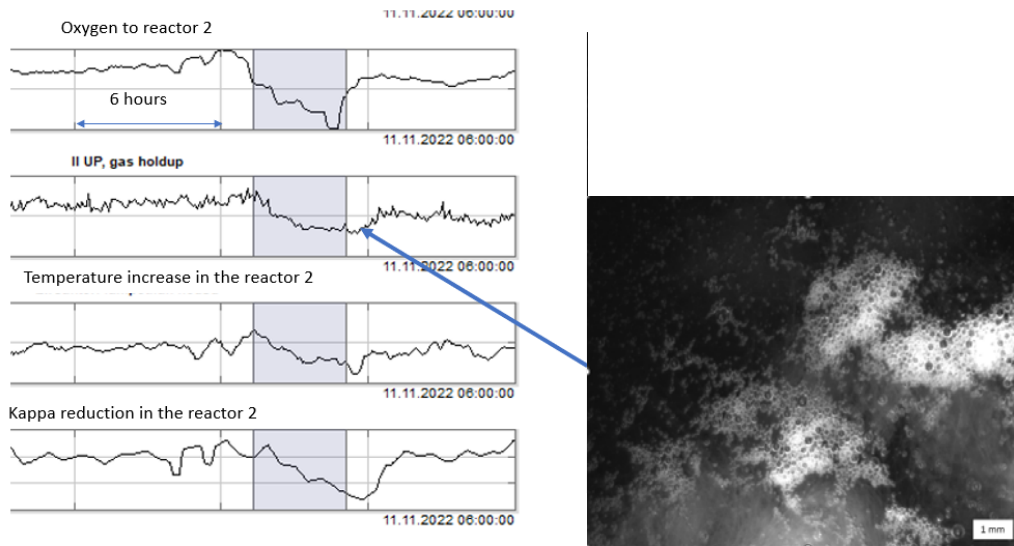


Figure 3. Effects of the oxygen charge in reactor 2.

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, (see Fig. 4).



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

It turned out that this mill is 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 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/t) in the process by measuring the oxygen content 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 O₂ reactor are connected (see Fig. 5). According to mass balance, the concentration of oxygen in the second reactor residual gas was 0 kg/t. Here could also be obtained information 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.

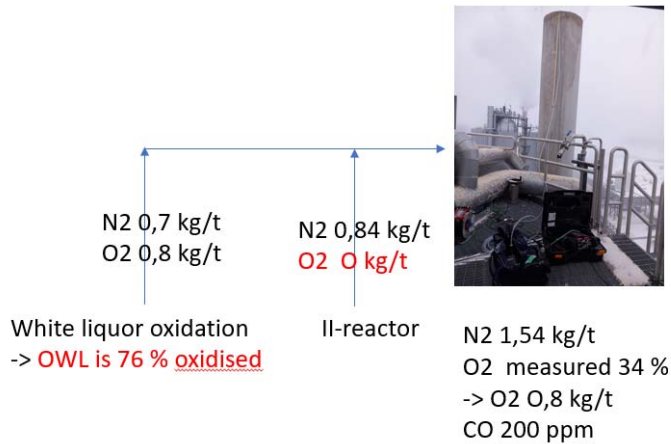


Figure 5. The mass balance of nitrogen and oxygen in the system.

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 (see Fig. 6). When the oxygen charge was increased by 3 kg/t 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.

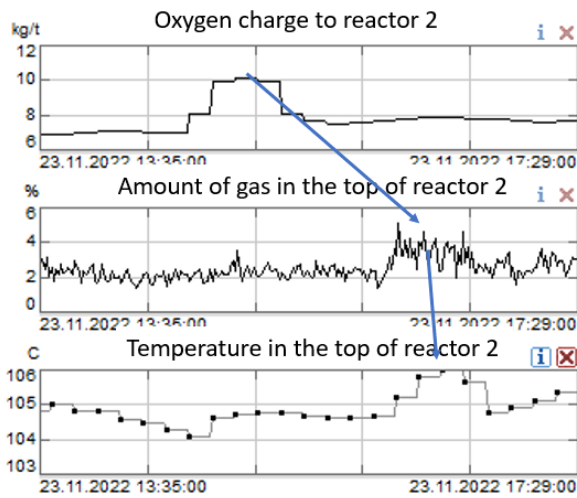
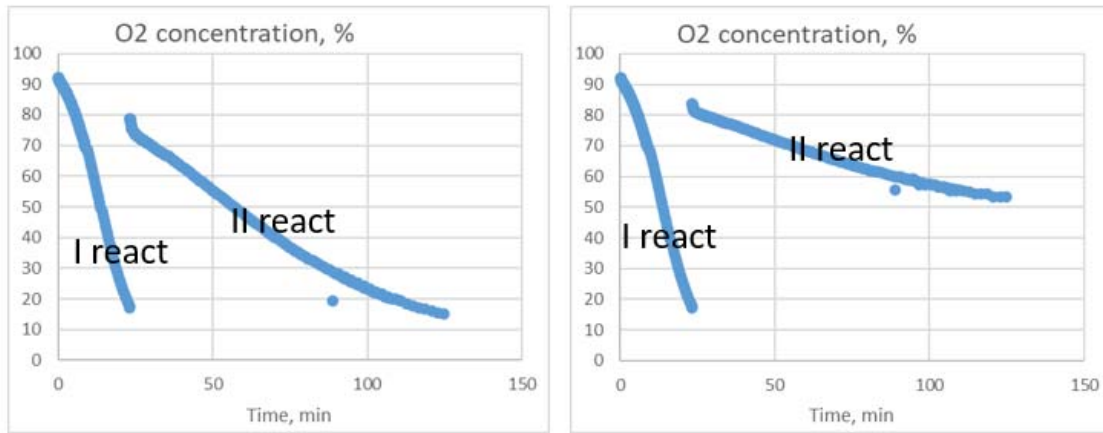


Figure 6. Effect of oxygen charge on the amount of residual gas and temperature in the top of the second reactor.

According to the oxygen delignification modeling, the following result for the concentration of dissolved oxygen in the reactors is obtained (see Fig. 7). When the oxygen charge to reactor 2 is increased by 3 kg/t, 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.



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

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 which 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 (see Fig. 8). This gives the possibility to gain new essential information from the process to be used in the modeling. The oxygen concentration directly indicates 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/t) can be calculated and the exact amount of oxygen consumed in the reactor is then also obtained.



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

The oxygen charge to reactor 1 was increased from 11 to 14 kg/t and at the same time the concentration of oxygen was measured from the top of the reactor and it increased from 15 %

to 55 % (see Fig. 9). At the same time, the temperature in the top of the reactor increased about 0.7 °C and the kappa decreased about 1.3 kappa units.

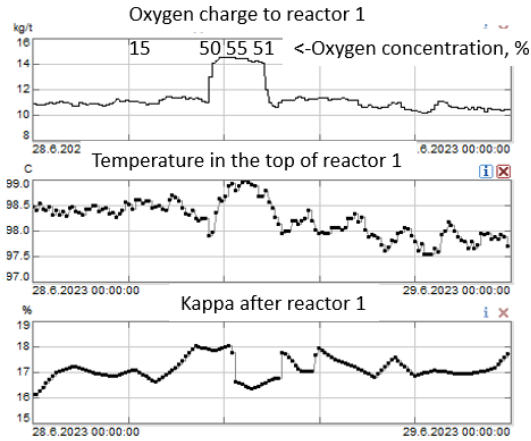


Figure. 9. Oxygen charge to reactor, kappa after reactor, oxygen concentration and temperature in the top of the reactor during the mill trial.

The model was fixed so that concentration of oxygen in the residual gas was set to 15 % in the top of the reactor and the kappa reduction after both reactors was the same in the model as observed in the mill (see Fig. 10).

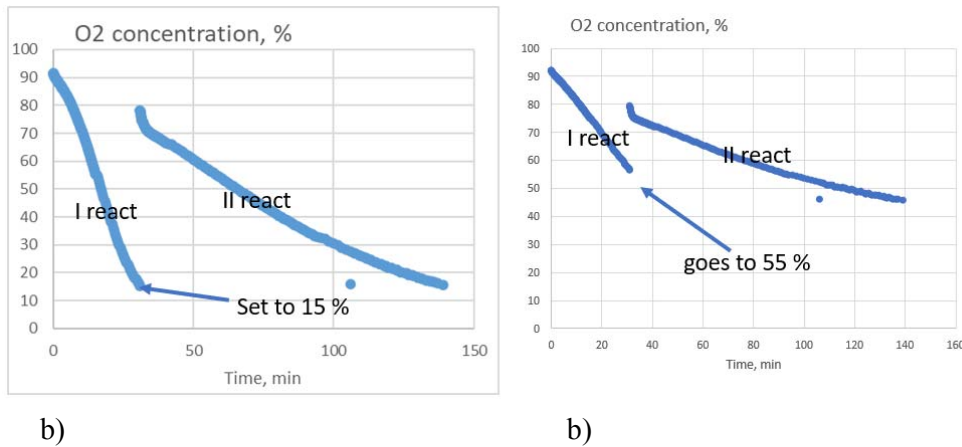


Figure 10. Concentration of oxygen in the reactors without oxygen charge increase (a) and with oxygen charge increase by 3 kg/t (b).

According to the modeling, a 3 kg/t charge increase to the first reactor should decrease the kappa after first reactor by 2.0 kappa units which is a little higher than that observed in the process. In addition, the oxygen concentration in the top of reactor 1 should rise to 55 %, which is 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 % (see Fig. 11). In addition, according to the modeling it should have risen to 47 % (see Fig. 12).

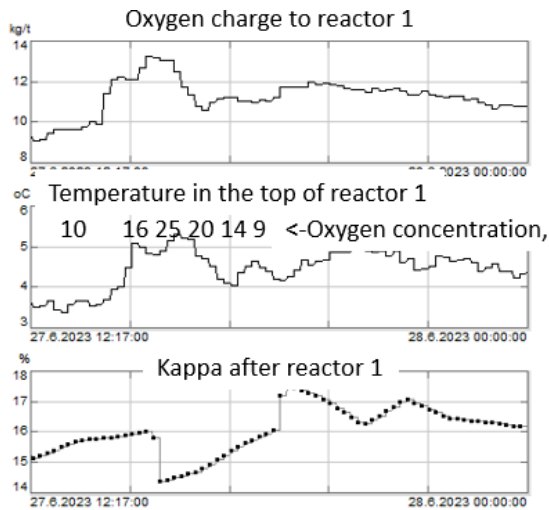
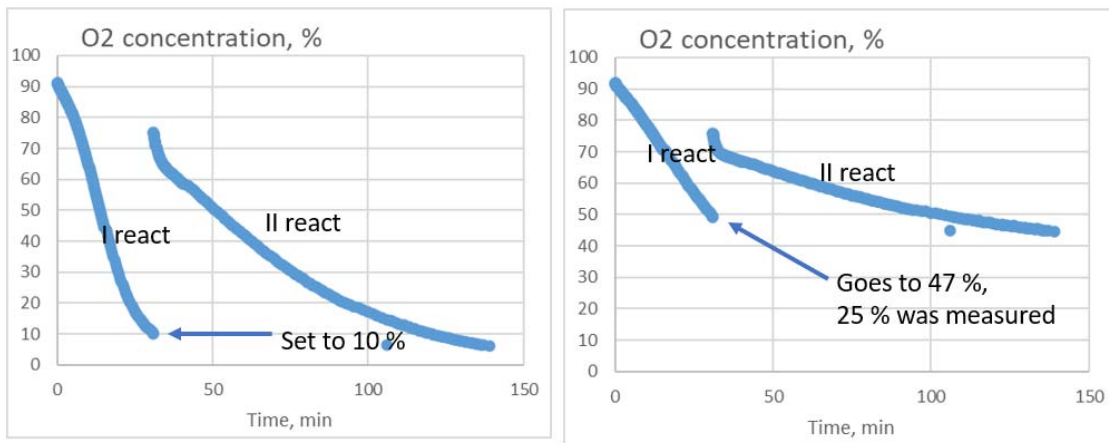


Figure 11. Oxygen charge to the reactor, kappa after reactor, oxygen concentration and temperature in the top of the reactor during the mill trial.



a)

b)

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

In the test undertaken in reactor 2, a similar result was obtained (see Fig. 13). The charge of oxygen was raised 3 kg/t, the temperature increased and the kappa decreased but the concentration of oxygen did not increase at all. This is very odd, since 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 nothing should happen.

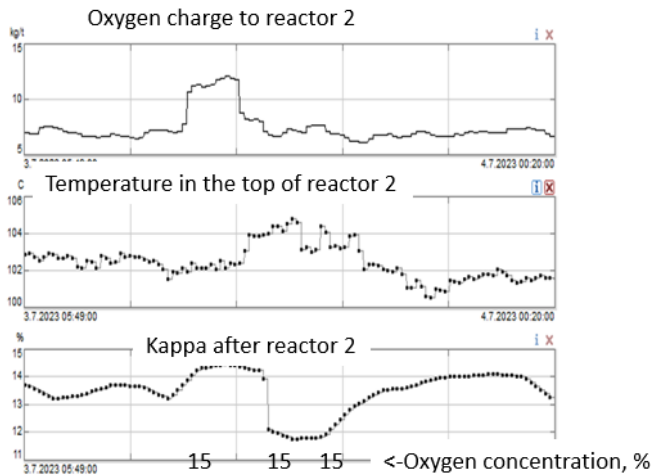


Figure 13. Oxygen charge to reactor, kappa after reactor, oxygen concentration and temperature in the top of the reactor during the mill trial.

One explanation for this is that some reason we did not collect a representative sample for the oxygen concentration measurement. Another interesting explanation is that there are heterogeneities in the reactor, i.e. the consumption of oxygen and/or distribution of oxygen gas in the reactor are not even and there are places where the concentration of oxygen is very small or zero. This seems to be the case especially in reactor 2. The motor in the oxygen mixer in reactor 2 is much smaller compared to reactor 1 which may explain this difference.

Effect of residual gases on the washing

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

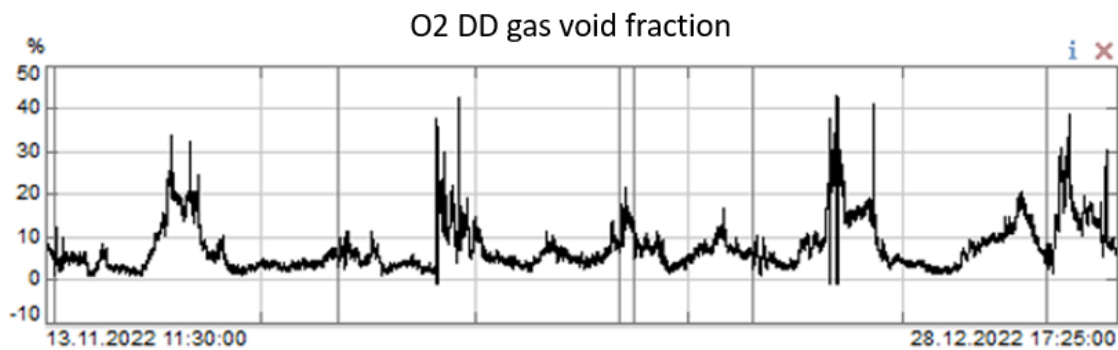


Figure 14. Gas content in the feed of the DD-washer measured with Echowise gas measurement.

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

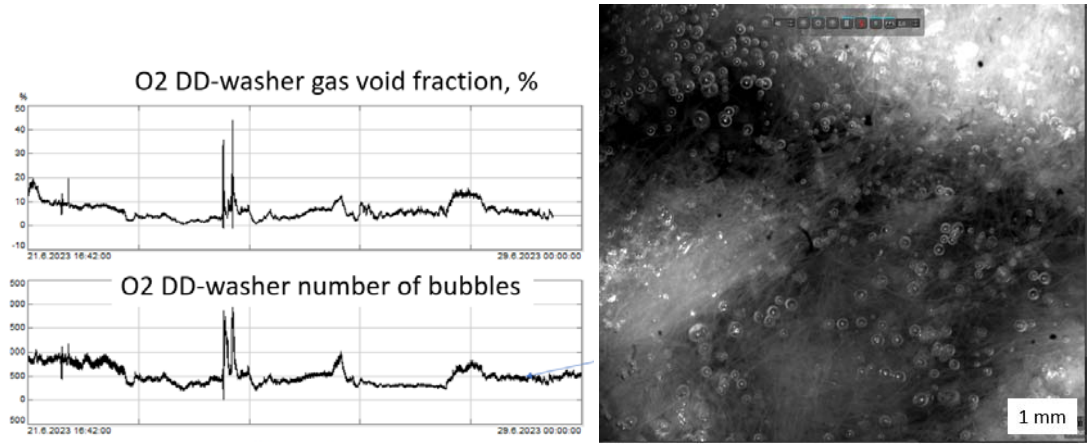


Figure 15. Gas content in the DD-washer feed pulp based on the Echowise and bubble imaging-based measurements and an example of the bubble image.

During the mill test the aim was also to gain information on how increasing the oxygen charge increases the gas content in the feed of the DD-washer (see Fig. 16). This information was not obtained, since the oxygen charge increased the temperature in the top of reactor 2, this increased vaporization of water and hence removal of gases from pulp and the gas content in the washer decreased.

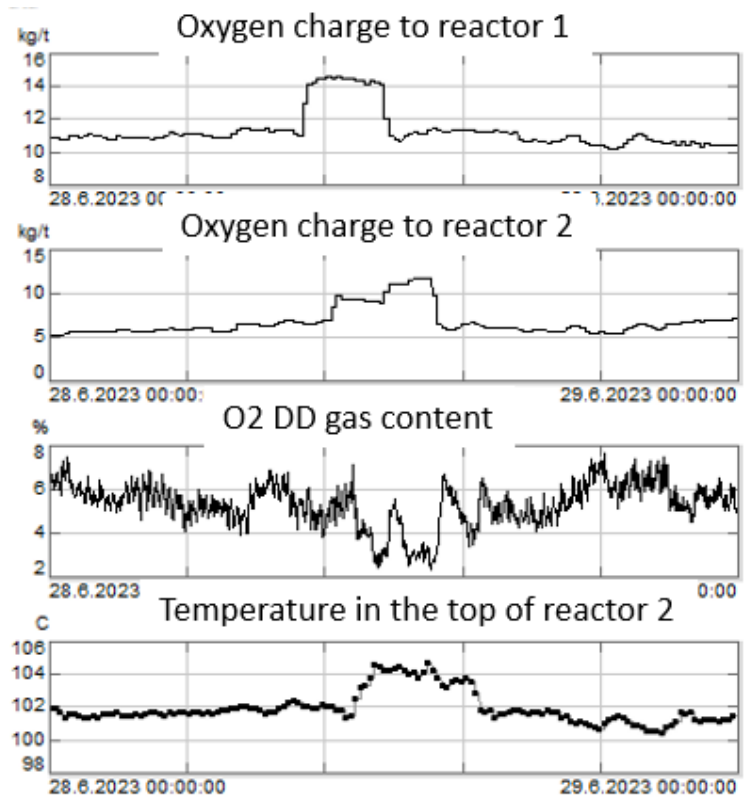


Figure 16. Effect of oxygen charge on the gas content in the feed of the DD-washer pulp and the temperature in the top of the second reactor.

The next appropriate questions are whether the new gas removal systems in the top of the reactors are working so well that oxygen charges could be increased and even so well that removal of residual gases by flashing is no longer necessary and process temperatures could be decreased.

Conclusion

In this paper, the modeling of real 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, e.g.:

- 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 much valuable information for the 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 sufficiently well. 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.

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