Impact of dissolved matter in the oxygen delignification stage

CAROLINE WILKE, NICLAS ANDERSSON, AND ULF GERMGÅRD

ABSTRACT: The presence of dissolved matter in the pulp suspension in an oxygen (O_2) delignification stage, consisting mainly of dissolved lignin, is normally considered to negatively affect the delignification rate due to the competing reactions between the fiber bound lignin and the lignin dissolved in the filtrate. Recirculated oxidized filtrate from the post-O_2 washing is usually considered to be less harmful to the delignification efficiency than unoxidized dissolved matter originating from the cooking stage. To develop a better understanding of the mechanisms of the dissolved matter's reactions and impact on the O_2 stage performance, laboratory oxygen delignification experiments with varying levels of unoxidized and oxidized dissolved matter were conducted.

The results showed that unoxidized dissolved matter had a negative impact on the delignification in the O_2 stage, whereas oxidized dissolved matter actually had a positive effect. The delignification efficiency of the laboratory experiments thus depends on both the amount of dissolved matter and its origin. The pulp viscosity decreased with increasing dissolved matter content irrespective of its origin but at higher COD levels; however, the viscosity drop was larger for the unoxidized dissolved matter. In terms of selectivity, the oxidized filtrate had a similar impact as additional NaOH charge. Both types of filtrates consumed hydroxide and the final pH decreased with increasing dissolved matter content. The final pH was significantly lower in the unoxidized filtrate experiments at higher COD levels, indicating a high reactivity between the unoxidized dissolved matter and the oxygen in the reactor.

Based on the results, further understanding is achieved about the relation between pre-O_2 washing performance and process configuration in an actual mill case, as well as the impact of dissolved matter on delignification. The importance of efficient removal of harmful unoxidized dissolved matter is verified, but the results also suggest that the oxidized dissolved matter that is recirculated from post-O_2 washing actually has a significant positive impact on the delignification and is not just a potential problem in case of carryover to the bleach plant. Subsequently, pulp washing efficiency is critical both pre- and post-O_2.

Application: The results from the study reinforce the importance of well-performing brownstock washing to minimize the carryover of unoxidized dissolved matter from the cooking stage. However, because the oxidized post-O_2 filtrate actually has a significant positive impact when recirculated, this can be exploited to improve delignification and reduce alkali consumption in the O_2 stage. Subsequently, optimum operation of pre- and post-O_2 washers will depend on the mill's washer configuration.

To accurately control the degree of delignification in a pulp mill fiber line, the most frequently used parameter is the fiber kappa number, especially in the beginning of the delignification, i.e., in the digester and the following oxygen (O_2) delignification stage. Studies [1-4] have shown, however, that the kappa number reduction is strongly influenced by the presence of dissolved matter in the pulp slurry, both in the cook and in the following O_2 delignification. This is clearly shown in the O_2 delignification stage, which was the target for this study. The dissolved matter generated in the digester and the O_2 stage is largely removed through washing of the pulp; however, an industrial washer is never 100% perfect. The remaining amount of dissolved matter in the pulp after washing is usually referred to as "wash loss" or "carryover," and it consists of both inorganic and organic dissolved matter. The carryover of dissolved matter is often characterized as the chemical oxygen demand (COD), and it is typically expressed as kg COD/ton of pulp. Good washing of the pulp before the O_2 stage minimizes the amount of unoxidized dissolved matter entering the O_2 stage with the pulp. Large carryover of unoxidized dissolved matter will reduce the O_2 delignification response and consume more oxygen and sodium hydroxide (NaOH) in the O_2 stage [5-10]. The higher chemical consumption is due to competing reactions between the dissolved lignin in the carryover and the lignin in the fibers [5,8,10]. Studies have shown that the alkali consumption by the dissolved matter can partly be counteracted for by adding more NaOH in the O_2 stage [7-10].

There are two kinds of dissolved matter that enter the O_2 stage: unoxidized dissolved matter originating from the digester and oxidized dissolved matter from post-O_2 washing. The degree of pre- and post-O_2 washing will not only deter-

mine the amount of dissolved matter carryover but also the distribution between unoxidized and oxidized dissolved matter [10]. According to previous studies, recirculated oxidized filtrate from post-O_2 washing is less harmful to the delignification efficiency than unoxidized filtrate originating from the cooking stage [5-9]. The unoxidized filtrate is more reactive than oxidized filtrate, and the competing reactions between dissolved lignin and the fiber bound lignin are thus more pronounced for the unoxidized filtrate. However, Miller et al. [10] claimed that oxidized dissolved matter was more harmful to the O_2 delignification stage than unoxidized dissolved matter due to its low content of alkali. All of the mentioned studies agree that dissolved matter indeed has an effect on the efficiency of the O_2 stage, but the size of the impact differs. Some of the studies [6,7,10] showed that small amounts of unoxidized dissolved matter could have positive effects on the O_2 delignification stage, while larger quantities always decreased the delignification. A study by Iijima and Taneda [5] indicated that oxidized dissolved matter was beneficial for delignification, but they only studied low COD levels, i.e., below 18 kg/ton. Miller et al. [9] showed that oxidized filtrate enhanced delignification at 130 kg COD/ton, but it was explained by the reactive nature of the pulp used. Bokström and Nordén [11] concluded that oxidized dissolved matter was beneficial for the viscosity-kappa number selectivity at COD levels up to 160 kg/ton.

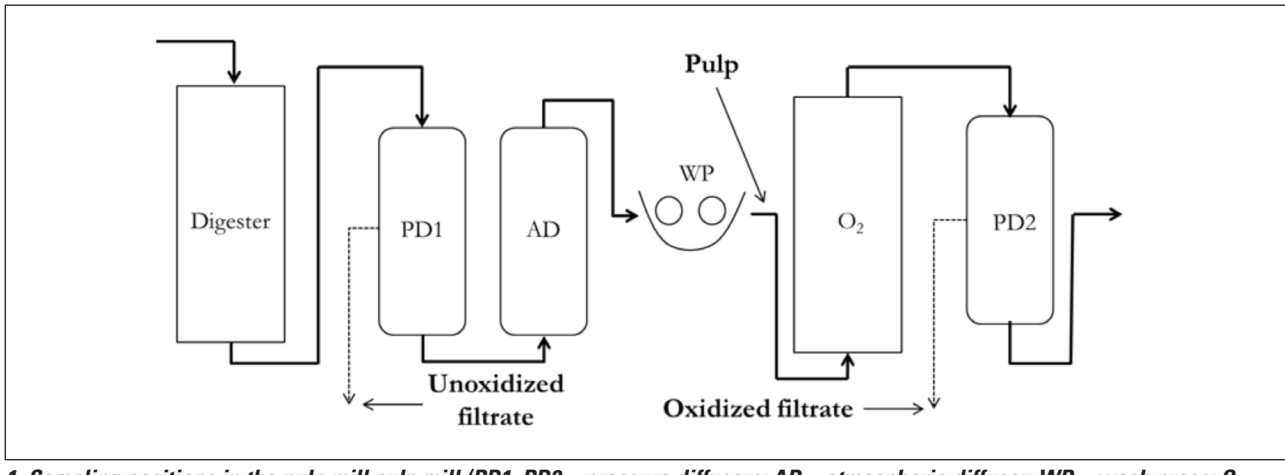
Hence, in the literature, there is no unanimous conclusion regarding the impact of dissolved matter, so the objective of this study was to further investigate the effects of carryover on the O_2 delignification responses. Three types of dissolved matter were used: unoxidized, oxidized, and mixtures of the two.

EXPERIMENTAL

This study was carried out in the laboratory on softwood kraft pulp taken from a Scandinavian pulp mill. As can be seen in **Fig. 1**, the pulp had been washed in a pressure diffuser (PD1) after the digester, followed by an atmospheric diffuser (AD) and a wash press (WP). The sampling position for the pulp was located at the screw after the wash press (WP). Filtrates from pre- and post-O_2 washing systems were also obtained from the mill. They were collected from the pressure diffusers before and after the O_2 delignification stage. The filtrate collected before the O_2 stage was assumed to contain primarily unoxidized dissolved matter, and filtrate collected after the O_2 stage was assumed to contain exclusively oxidized dissolved matter. The sampling positions were chosen so that the pulp sample represented the actual pulp entering the O_2 stage, whereas the filtrate samples were chosen to allow for the examination of the effect of the different types of dissolved matter. In reality, the pulp slurry entering the O_2 delignification stage contains both unoxidized and oxidized dissolved matter due to the recirculation of filtrates in the different washing stages. In the first set of experiments, the pulp samples were thoroughly washed in the laboratory, whereas in the second set of experiments, the pulp samples were unwashed. The sampling positions and the properties of the collected samples are depicted in Fig. 1 and **Table I**.

The O_2 delignification experiments were conducted in 2.5 L autoclaves immersed in a polyethylene glycol bath, and the laboratory conditions are listed in **Table II**. Filtrate was added to the pulp samples in most of the experiments in order to simulate various levels of dissolved matter. The filtrates were diluted with deionized water to achieve different levels of COD. In order to reach certain COD levels, the dilution for the unoxidized filtrates was much larger, thus causing their pH values to decrease more compared to the oxidized filtrates.

Some preparatory experiments were conducted in order to exclude any impact from suspected disturbance factors. The presence of metal ions can affect the pulp viscosity [12]. Oxygen delignification experiments were performed with ethylenediaminetetraacetic acid (EDTA), which acts as a chelating agent to negate the effect of metal ions. The results showed that there was no significant difference in pulp viscosity between the experiments with and without added ETDA. Furthermore, hydrogen sulfide ions are known to affect negatively pulp viscosity, and to some extent also delig-



1. Sampling positions in the pulp mill pulp mill (PD1, PD2 = pressure diffusers; AD = atmospheric diffuser; WP = wash press; O_2 = oxygen delignification).

	Viscosity, dm^3/kg	Kappa Number (of clean pulp)	COD, g/L	pH
Experiments with washed pulp (I)	**1247**	**26.8**	**0.0**	
Unoxidized filtrate			120.0	12.5
Oxidized filtrate			15.5	10.7
Experiments with unwashed pulp (II)	**991**	**27.3**	**56.5**	**10.5**
Unoxidized filtrate			135.5	12.1
Oxidized filtrate			35.0	11.8

I. The properties of the original samples used in the experiments.

Pulp, g	50
Consistency, %	10
COD, kg/ton pulp	25-360
NaOH, %	2
$MgSO_4$, %	0.2
Pressure, bar	5
Temperature, °C	90
Time, min	120

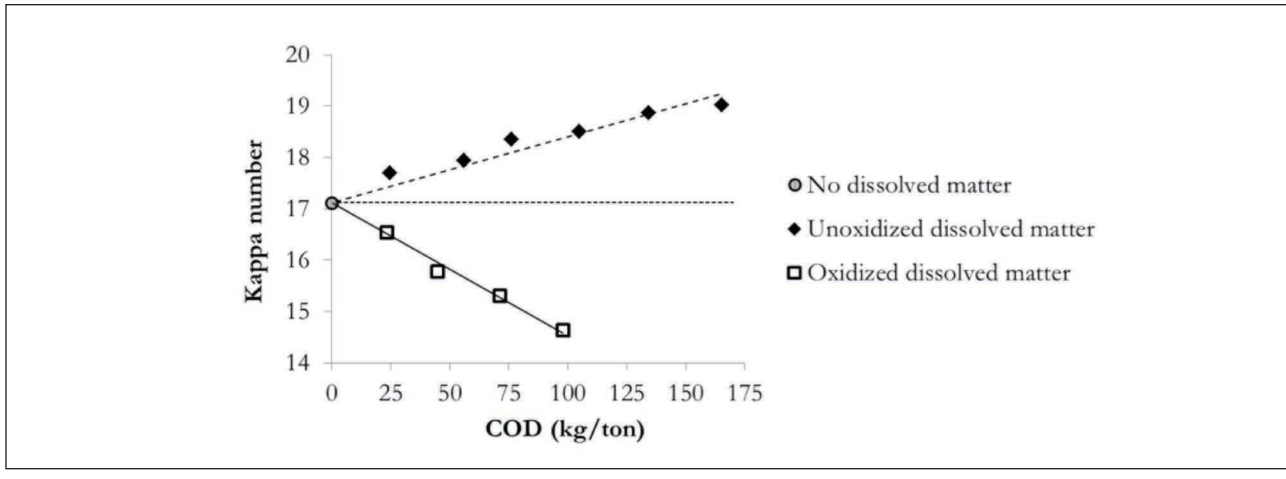
II. Laboratory conditions used, where the charges are calculated on dry pulp.

nification [13]. In order to ascertain that the hydrogen sulfide ions did not impact the experiments, a comparison was made between a thoroughly aerated unoxidized filtrate and the non-treated one. The differences in kappa number and pulp viscosity after the O_2 delignification stage were marginal. The hydrogen sulfide ion concentration was also determined by titration (SCAN-N 31:94 "Hydrogen sulfide ion concentration") and the very low level confirmed the observations (0.06 M HS^- for the raw unoxidized filtrate). Furthermore, the COD was measured before and after aeration, again showing no significant difference.

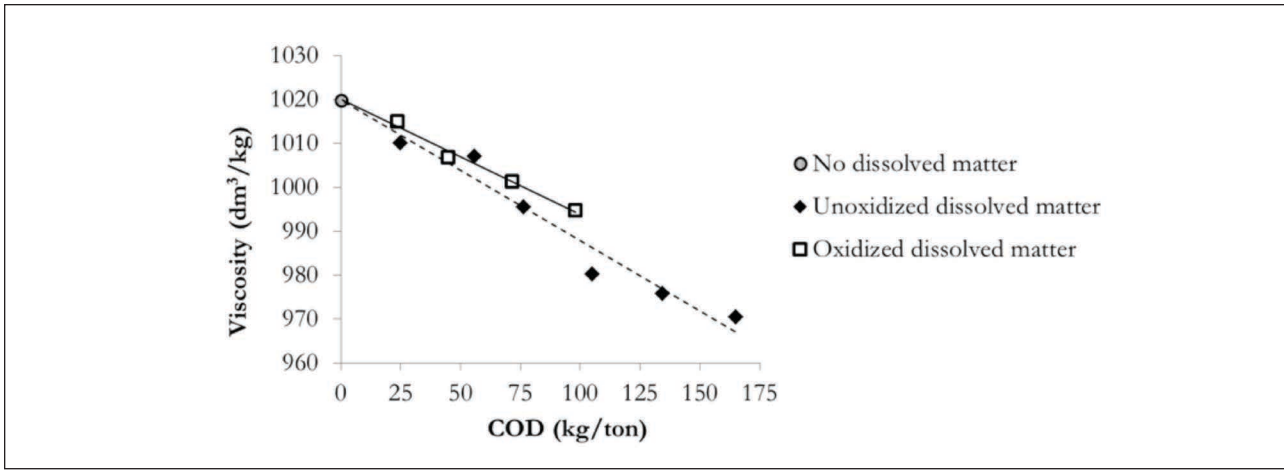
RESULTS AND DISCUSSION

Experiments with washed pulp

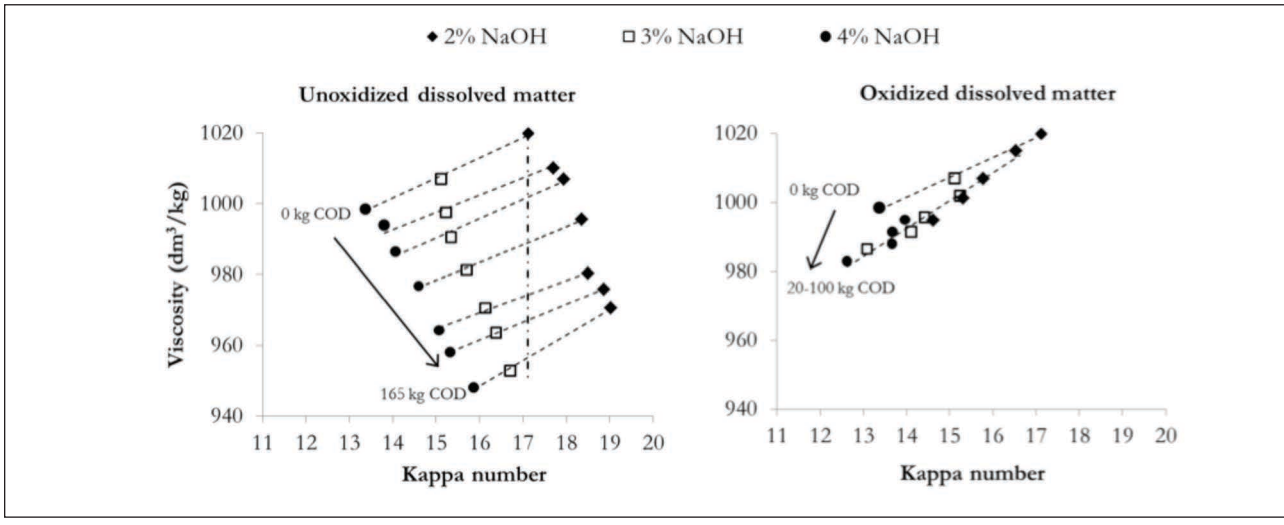
The first set of experiments were done on a well-washed pulp sample (pulp I in Table I). Laboratory O_2 delignification experiments were conducted on this pulp in order to investigate how varying contents of unoxidized and oxidized dissolved matter influenced lignin removal during the O_2 delignification. In **Fig. 2**, the resulting fiber kappa numbers after the O_2 delignification experiments are plotted vs. the COD in the samples prior to the O_2 stage. As can be seen, the experiment with the well-washed pulp sample (i.e., without dissolved matter) reached a kappa number of 17.1. It is evident that the presence of unoxidized dissolved matter had a relatively large negative impact on the delignification, with resulting kappa numbers up to 19.0 at the highest COD levels studied (i.e., 165 kg/ton). This result is in agreement with earlier studies [6-9]. The presence of oxidized filtrate, on the other hand, had a positive impact on delignification, and in this case, the resulting kappa number after the O_2 stage was down to 14.5



2. Fiber kappa number vs. chemical oxygen demand (COD) with unoxidized and oxidized filtrates.



3. Viscosity vs. COD with unoxidized and oxidized filtrates.



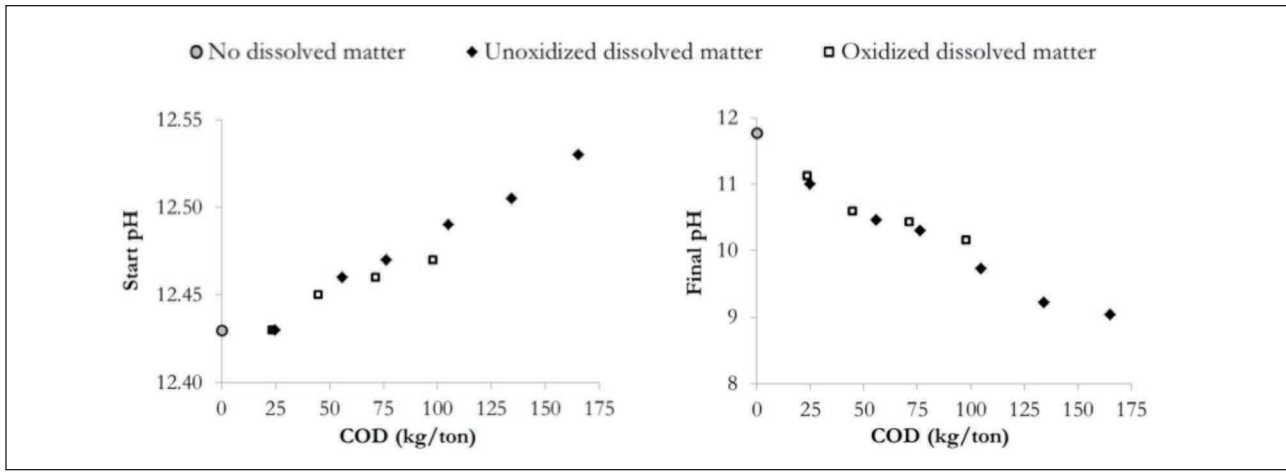
4. Viscosity vs. kappa number at different sodium hydroxide (NaOH) charges with unoxidized (left) and oxidized (right) filtrates.

at the highest COD level. Small positive effects of oxidized dissolved matter have previously been indicated by several investigators [5,9,11], but none have observed the significant impact presented by the current study.

The corresponding pulp viscosities after the O_2 delignification stage vs. COD were found to decrease in a similar manner for both types of filtrates, as shown in **Fig. 3**. The results with the unoxidized dissolved matter are somewhat scattered, but still indicated that the viscosity loss was slightly more than that of the oxidized dissolved matter.

The effect on selectivity by the two types of filtrates was investigated by additional experiments with 3% and 4% NaOH charges, and the results are shown in **Fig. 4**. It is evident that the additional NaOH charge yielded considerably lower kappa numbers, while the viscosities were rather moderately affected. The relative effect on delignification and viscosity degradation was principally independent of the COD level and was also independent of the type of dissolved matter (i.e., unoxidized or oxidized) with respect to the additional NaOH charge. However, as noted earlier, the presence of unoxidized dissolved matter itself had a very negative impact on the selectivity in the O_2 stage. One can also note that the addition of oxidized dissolved matter actually influenced the O_2 delignification in a similar way as a higher NaOH charge, resulting in just a slightly lower viscosity compared to the reference case (0 kg COD). The dash-dotted line in the left subplot, for unoxidized dissolved matter, can be used to approximate the additional NaOH charge required to counter effect the COD charge compared to the reference case with no COD. A rough assessment is that an additional 0.5% NaOH charge is required per 100 kg COD, or 0.05 kg NaOH per kg COD. Furthermore, note in the right subplot, for oxidized dissolved matter, that all points with any COD charge are on the same trend line. However, the symbols used are the same as for the left subplot, and for any NaOH charge, an increase in the COD charge will lower both kappa number and viscosity.

It has previously been suggested by investigators [5,8-10] that the alkali contribution from the filtrate could have a pos-



5. Start pH (left) and final pH (right) vs. COD with unoxidized and oxidized filtrates.

itive effect on the delignification and a negative effect on the pulp viscosity. However, the additional NaOH contribution from the filtrates at 100 kg COD/ton (determined with SCAN-N 33:94 "Residual alkali [Hydroxide ion content]") was only 0.14% NaOH (on pulp) for the unoxidized filtrate and 0.09% for the oxidized one. Such small NaOH contribution compared to the NaOH charges shown in Fig. 4 is thus not likely to be the reason for the differences observed for delignification and pulp viscosity.

The filtrate pH was determined at 25°C before and after the laboratory O_2 delignification, and the results can be seen in **Fig. 5**. The starting pH values were measured after the addition of 2% NaOH. The differences in initial pH were very small over the studied COD range due to the low alkali contribution from the filtrates, which was about 0.1% NaOH, as previously described. However, the differences in pH after the O_2 delignification were substantial, indicating that dissolved matter consumed a significant amount of alkali. The large consumption could have explained the poor delignification observed for the unoxidized filtrates, but not the beneficial delignification for the oxidized ones. Subsequently, according to the present study, hydroxide consumption and pH levels alone cannot explain the difference in delignification observed for unoxidized and oxidized matter, at least up to 100 kg COD/ton.

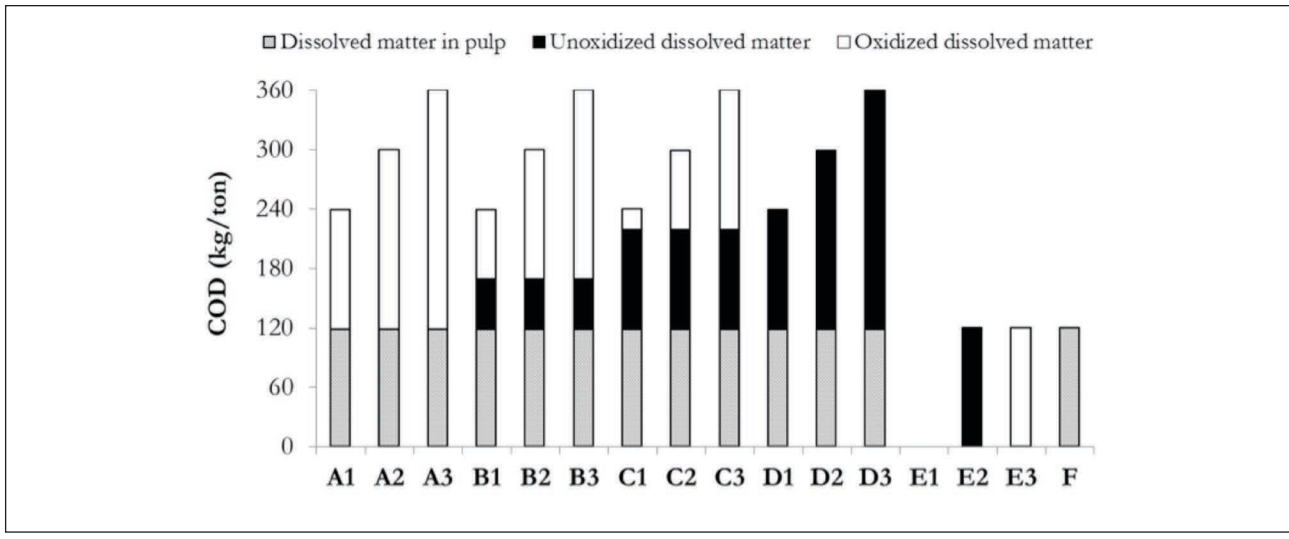
It has previously been reported [10] that unoxidized dissolved matter generates more organic acids than oxidized dissolved matter, thus resulting in lower final pH. The results in these experiments indicated, however, that the two types of filtrates have similar final pH up to 100 kg COD/ton. Other studies [8,9] have stated that oxidized dissolved matter had a higher carbon dioxide evolution, which resulted in a higher NaOH consumption. It may be that the larger organic acid generation from the unoxidized dissolved matter and the higher carbon dioxide evolution from oxidized dissolved matter affected the filtrates equally, thus resulting in similar final pH.

Experiments with unwashed pulp

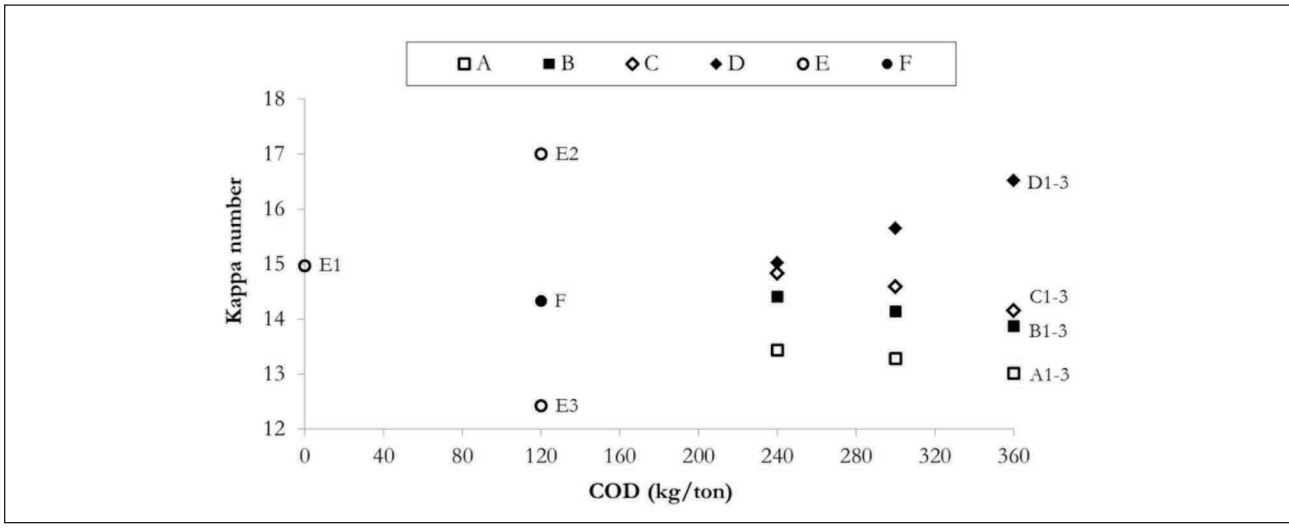
In order to ensure that the results in the previous experiments were not affected by the laboratory pulp washing, a further set of experiments was performed using unwashed pulp (pulp II in Table I), i.e., pulp sampled at the mill's wash press outlet in Fig. 1 (at approximately 30% consistency). Five series of experiments were performed where the additions of unoxidized and oxidized filtrates were altered according to **Fig. 6**. The unwashed pulp had a COD contribution of 120 kg/ton (sample F). The actual O_2 delignification feed at this mill corresponded to sample A3 at the time of collecting the pulp and filtrate samples. A previous study using an inline sensor for measurement of dissolved lignin in the O_2 delignification stage inlet at the same mill showed that the COD typically varied between 90 and 390 kg COD/ton [4].

The pulp (II) sample used in these experiments had a significantly lower pulp viscosity than the pulp (I) used in the experiments (see Table I). According to Miller et al. [9], lower pulp viscosity indicates that the fiber structure is more open and lignin reaction sites more accessible, thus making the pulp more reactive. Some pulp (II) was therefore washed and oxygen delignified for comparison. Note that sample E1 did not contain any dissolved matter; hence, the COD is 0 kg/ton in Fig. 6.

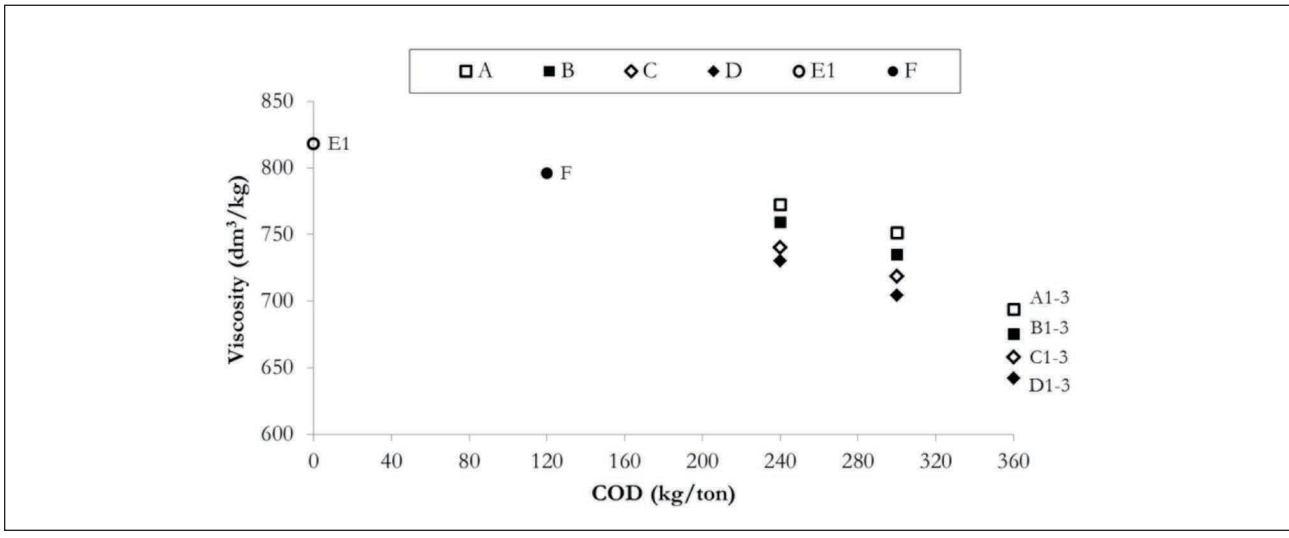
The kappa numbers after oxygen delignification of pulp (II) are shown in **Fig. 7**. The two washed samples with added unoxidized and oxidized filtrates (E2 and E3) resulted in kappa numbers that were in agreement with the previous studies with washed pulp samples. The kappa number for the sample containing only unwashed pulp (F) was 0.5 units lower than that of the washed sample (E1), indicating that slightly more than half of the pulp's dissolved matter was oxidized. Note that the oxygen delignification feed consisted of a mixture of unoxidized and oxidized filtrates, and this distribution varies from one mill to another mill. Results by Miller et al. [10] are in agreement with this, as the authors showed that 70%–92% of the dissolved matter entering the O_2 stage consisted of oxidized dissolved matter. Note that this percentage was the actual O_2 delignification feed, thus including the dilution with the oxidized filtrate as well.



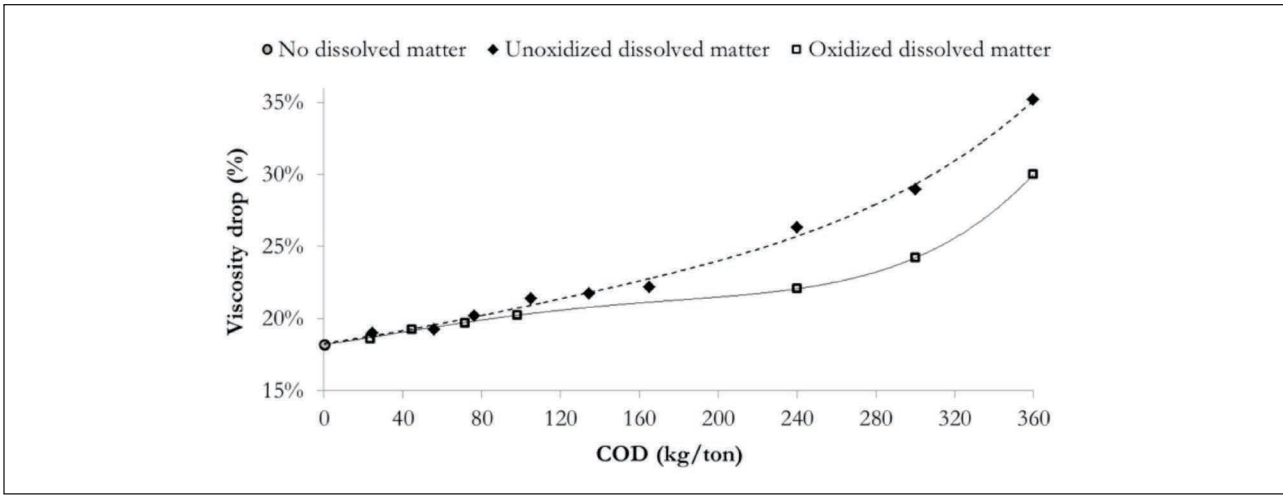
6. The amount and origin of COD in the experiments with unwashed pulp.



7. Kappa number vs. COD for the different experiments.



8. Viscosity vs. COD for the different experiments with unwashed pulp.



9. Viscosity drop vs. COD with unoxidized and oxidized filtrates. Pulp (I) is 0–165 kg COD/ton and pulp (II) is 240–360 kg COD/ton.

When comparing series A and D, it is clear that delignification benefited from increasing amounts of oxidized filtrate. The highest delignification was for sample A3, which resulted in a kappa number 1.5 units lower than that for the unwashed reference sample (F). From the kappa numbers of the D series, it is again evident that unoxidized filtrate had a negative impact on the delignification, which was more pronounced at high COD levels. Kappa numbers from the B and the C series further confirmed that the effect of additional unoxidized dissolved matter is always negative for delignification, while the effect of additional oxidized dissolved matter is always positive.

The results showed that good washing before the O_2 stage is important in order to remove as much as possible of the unoxidized dissolved matter. Oxidized filtrate is currently being used for dilution before the O_2 stage in mills, and the results indicate that this is very beneficial in order to exploit the positive effect of oxidized dissolved matter, at the same time minimizing the impact of any carryover of unoxidized dissolved matter.

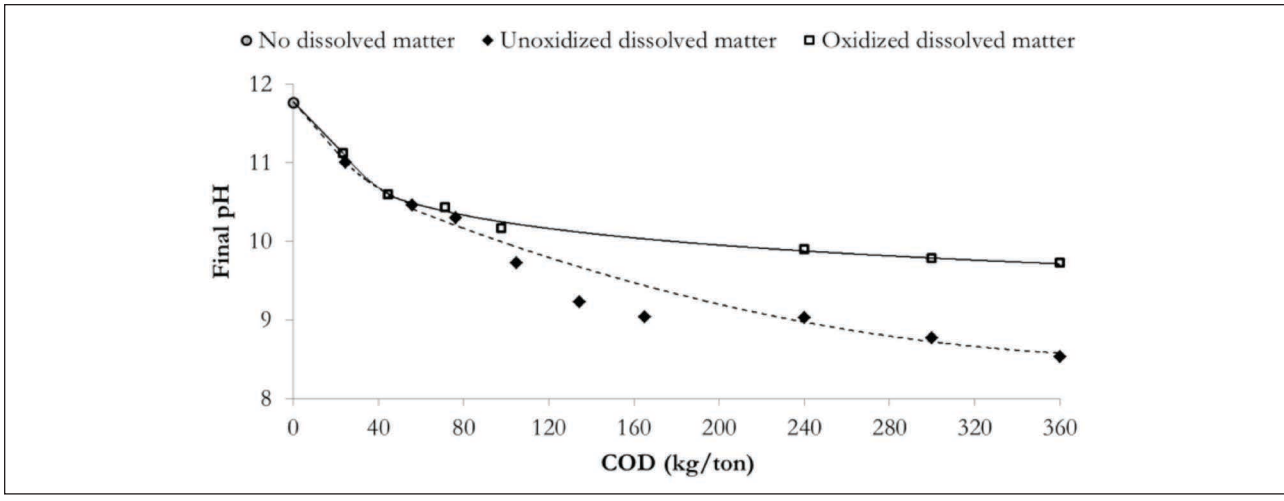
The pulp viscosities for series A to D, along with the reference samples E1 (washed pulp) and F (unwashed pulp), are shown in **Fig. 8**. It can be seen that the viscosity decrease was more pronounced at higher COD levels. It is also evident that there was a difference between unoxidized and oxidized dissolved matter that was not observed at lower COD levels in the previous experiments with washed pulp (I).

Since the starting viscosity prior to the O_2 stage was much lower for this pulp (pulp [II]) compared to the experiments with well washed pulp (pulp [I]), the percentage decrease of pulp viscosities is a better way to compare the results; this is shown in **Fig. 9** for comparison. All of the viscosities for the experiments with pulp (I) are shown, but only series A to D with pulp (II) are shown. The viscosity drop due to dissolved matter appeared rather moderate up to around 200 kg COD/ton, i.e., less than 5% relative; furthermore, the impact of unoxidized and oxidized filtrates was very similar. However, the viscosity decrease was more notable at the higher COD values, i.e., above 200 kg/ton, and the unoxidized filtrate showed a more detrimental impact than the oxidized filtrate.

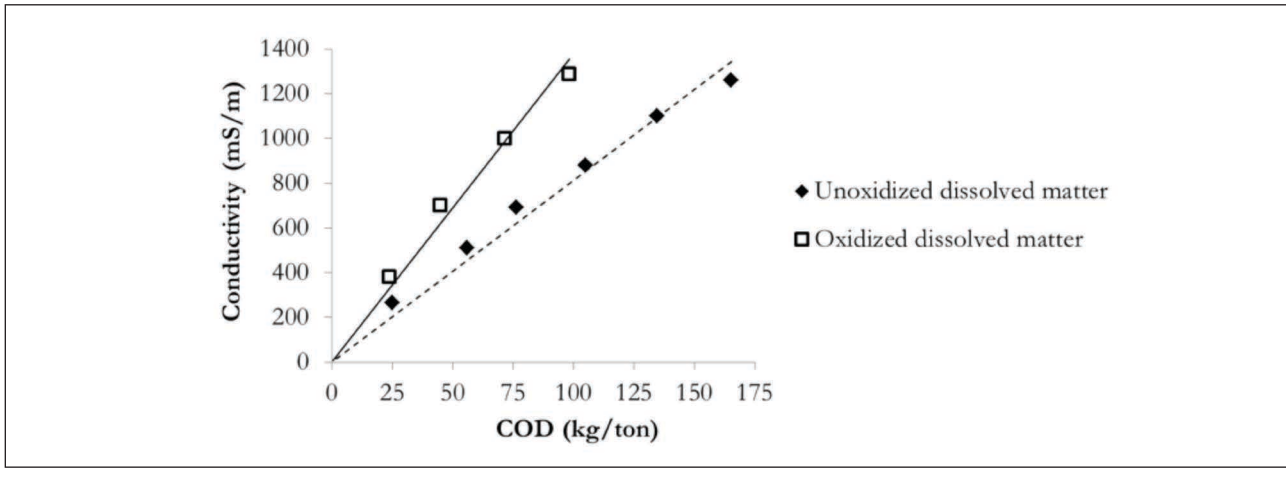
According to **Fig. 10**, there was a significant difference between the final pH values for additions of unoxidized and oxidized filtrates that were not observed in the experiments with pulp (I) using lower levels of oxidized dissolved matter. The final pH values for A and D series using pulp (II) are used for the comparison. It can now be seen that the unoxidized dissolved matter consumed significantly more hydroxide than the oxidized filtrate above 100 kg COD/ton. This is in agreement with a previous report [10], which stated that unoxidized dissolved matter has a higher chemical demand than oxidized filtrate, thus consuming more hydroxide and oxygen. Due to the dissolved matter, competing reactions between fiber-bound lignin and dissolved lignin occur, and hydroxide and oxygen are free to react with both types of lignin [7,8,10]. These reactions lower the delignification rate, and the poor delignification for the unoxidized filtrate could partly be explained by this.

Previous reports by Miller et al. [10] suggested that the viscosity loss was only significant when the final pH was below 9.7. The final pH for the oxidized filtrates remained at or above 9.7 at all COD levels, whereas the final pH dropped below this pH at COD levels above 100 kg/ton for the unoxidized filtrates. It can be seen in Fig. 9 that the viscosity drop was larger above 100 kg COD/ton for the unoxidized filtrate, which thus is in agreement with Miller et al. [10].

The oxidized filtrate contains a larger amount of carbonate ions than the unoxidized filtrate because of the previous exposure to oxygen, where dissolved organic matter was oxidized to carbonate [8,9]. A higher carbonate concentration will shift the equilibrium in Eq. (1) to the right, thus producing not only more bicarbonate or carbon dioxide, but also more NaOH. According to Miller et al. [8], the carbonate ion is a weaker base than the hydroxide ion and the rate would hence be hindered. However, the pulp delignification is significant-



10. Final pH vs. COD with unoxidized and oxidized filtrates. Pulp (I) is 0–165 kg COD/ton and pulp (II) is 240–360 kg COD/ton.



11. Conductivity vs. COD with unoxidized and oxidized filtrates.

ly improved with oxidized dissolved matter, and the presence of the carbonate ion could partly explain these results, as NaOH could potentially be produced. The carbonate ion could also act as a pH buffer in the oxidized filtrate [13], thus explaining the higher final pH compared to the unoxidized filtrate.

$$CO_3^{2-} + H_2O \leftrightarrow HCO_3^- + OH^- \leftrightarrow CO_2 + 2\ OH^- \qquad (1)$$

To assess the ionic strength of the respective filtrate, conductivity was measured. The results are plotted in **Fig. 11**. As expected, the oxidized filtrate had a substantially higher conductivity, and subsequently higher ionic strength, than the unoxidized filtrate. The difference in ionic strength or alkalinity could explain part of the differences in delignification and viscosity seen for the unoxidized and oxidized dissolved matter. Subsequently, the use of end pH alone may have limitations for an optimal O_2 stage control.

As described in the experimental section, tests comparing the addition of aerated and non-aerated unoxidized filtrates were made in order to rule out any impact of HS^- ions. However, another potential impact of sulfur species is $S_2O_3^{2-}$ ions, since they can be oxidized further to SO_4^{2-} in an O_2 stage and thus consume NaOH. The thiosulfate content of the filtrates was not determined in the study, but in case the content was higher in the unoxidized filtrate than in the oxidized one. This can also partly explain the relatively higher NaOH consumption thereof and the subsequently lower degree of delignification observed for experiments using filtrates with unoxidized dissolved matter. Further experiments are, however, required to confirm this hypothesis.

CONCLUSIONS

- Unoxidized and oxidized dissolved matter had different impacts on the delignification in a laboratory O_2 stage. Subsequently, the impact depended on the relative amount of these two types of filtrates in the pulp slurry. Unoxidized dissolved matter had a negative impact on the delignification, while oxidized dissolved matter had a positive impact.
- With the conditions employed in the laboratory experiments, any contribution of NaOH from either type of dis-

solved matter, unoxidized or oxidized, only had a very small impact on the delignification. This observation could not explain the positive impact due to oxidized filtrate nor the negative impact due to unoxidized filtrate.
- Both unoxidized and oxidized dissolved matter had a negative impact on the pulp viscosity. At higher COD levels, the unoxidized dissolved matter is more negative than the oxidized one.
- Both unoxidized and oxidized dissolved matter consumed hydroxide and the final pH after the O_2 stage decreased. At higher COD, the final pH when using unoxidized filtrate was significantly lower than that when using oxidized filtrate.
- The relative impact on delignification and viscosity degradation (i.e., selectivity) when using oxidized filtrate was very similar to that of using a higher NaOH charge. When using unoxidized filtrate, the selectivity was significantly impaired since the degree of delignification was low and the viscosity drop high.
- Good pre- and post-O_2 washing is important for high delignification and low chemical consumption, both in the O_2 stage and the subsequent bleach plant. The harmful unoxidized dissolved matter should therefore be removed as much as possible before the O_2 stage. The industrial standard to recirculate post-O_2 washing filtrate means the beneficial effect of the oxidized dissolved matter is utilized.
- Differences in the ionic strength of the unoxidized and oxidized dissolved matter in the filtrates may be related to their different impacts on delignification. It follows that using terminal pH for NaOH charge control may have some limitations for an optimal O_2 stage control at the industrial scale. **TJ**

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ABOUT THE AUTHORS

In a previous study, we showed that dissolved lignin in the first chlorine dioxide stage consumed, per kappa number, approximately the same amount of bleaching chemicals as fiber-bound lignin. The aim of the current study was to investigate whether this was also the case in the oxygen (O_2) delignification stage.

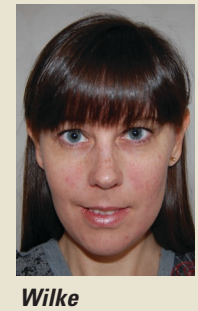
Many studies have been carried out on O_2 delignification over the years, and the results of these studies differ significantly with respect to the impact of dissolved matter. None of the previous studies has shown the same effects that we presented in our study, and our results are therefore unique.

The biggest challenge was to decide how to simulate, in a laboratory, the complex chemical reactions that take place in a commercial O_2 delignification stage, where temperature, pH, and dissolved matter influence each other in a very dynamic manner. We tried to solve this problem by using industrial filtrates from only one kraft pulp fiber line.

For us, the most interesting result was that the two different types of dissolved matter into the O_2 delignification stage had opposite effects on delignification. Despite different delignification, the pulp viscosities were affected similarly by unoxidized and oxidized dissolved matter.

For mills, good pre-O_2 washing is important for high delignification in the O_2 delignification stage, so it is important to maximize the washing efficiency. Recirculation of post-O_2 washing filtrate has beneficial effects due to the oxidized dissolved matter, so optimizing the recirculation could potentially increase the delignification.

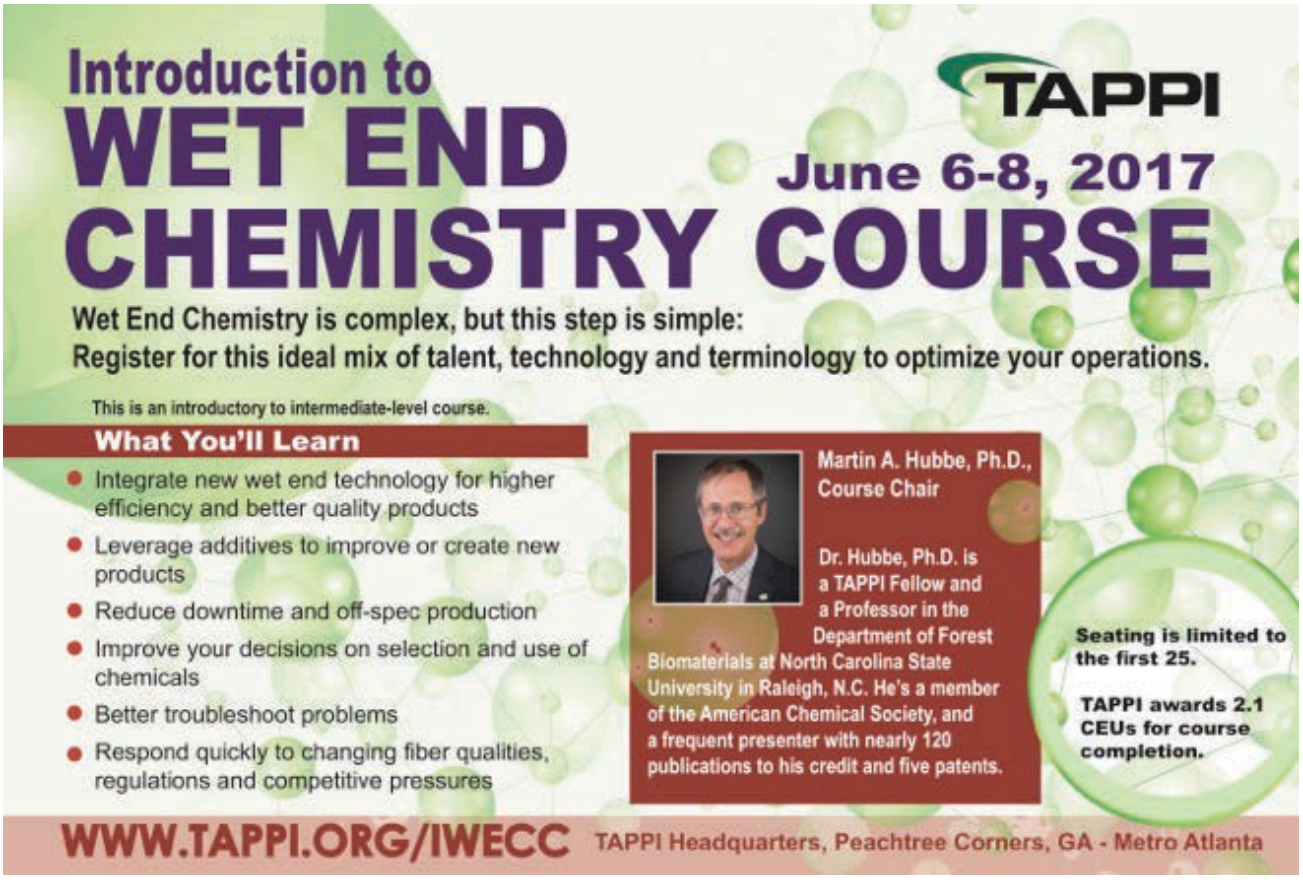
In the future, we plan to further investigate the effects of carbonate ions and thiosulfate ions in the O_2 delignification stage.



Wilke

Andersson

Germgård

Wilke is development engineer at BTG Instruments AB, Säffle, Sweden, and post-graduate student at Karlstad University in Karlstad, Sweden. Andersson is head of R&D and Business Development at BTG Instruments AB, Säffle, Sweden. Germgård is professor of Pulp Technology at Karlstad University, Karlstad, Sweden. Email Wilke at caroline.wilke@btg.com.

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