

Nonwood black liquor

Combustion properties of black liquors from alkaline pulping of wheat straw and reed canary grass

Jarmo Louhelainen, Raimo Alén, and Zhinan Feng

ABSTRACT

Combustion of black liquor after evaporation in the recovery furnace to recover energy and cooking chemicals is becoming a more attractive technique for nonwood pulping. However, due to the high viscosity of nonwood-based liquors, they require heat treatment before combustion to reduce viscosity. In this study, we investigated the combustion properties of black liquors from soda-anthraquinone (AQ) pulping of wheat straw and soda-AQ and kraft pulping of reed canary grass as such, and after heating at 190°C for 30 min, in a laboratory furnace at 800°C by a single droplet technique. In addition to swelling, the duration of different burning stages (devolatilization and char burning) was determined for each liquor. Finally, to develop rapid methods for predicting the combustion behavior of black liquor, adequate correlations between the FTIR (Fourier transform infrared) spectral data (indicating chemical composition) and the combustion properties of the studied black liquors were explored.

Application: Understanding the changes in combustion properties and FTIR spectrum of nonwood black liquors during heating leads to better control methods for black liquor recovery.

The worldwide capacity of nonwood pulping (nearly 11% of total pulping capacity in 1997) has increased faster than that of wood pulping [1]. Most nonwood pulp is produced in China, corresponding to more than 70% of the global production of nonwood pulp in 1993. While wood-utilizing pulp mills recover the cooking chemicals and energy from black liquor, most nonwood-utilizing pulp mills discharge their black liquors to receiving waters [2, 3]. In 1997, only 0.8% of the caustic soda used in the production of wheat straw pulps was recovered in China and about 4.5 million metric tons of organic pollutants (COD) were discharged [3]. This loading is gradually leading to serious environmental problems, but the possibilities for more efficiently recovering the cooking chemicals have emerged in recent years. One difficulty has been the small size of the mills, which has made it impossible to establish economical processes to recover cooking chemicals. This has also resulted in the increasing trend to construct bigger mill units instead of conventional small-scale mills [1, 3].

Combustion of the evaporated black liquor, as performed in wood-based pulp mills, has proven to be the most applicable method for nonwood-based mills to recover cooking chemicals and energy. However, due to the higher polysaccharide and silica (silicon dioxide) contents, evaporation of nonwood black liquors to high dry solids content needed for combustion is more difficult compared to that of wood black liquors. The polysaccharide constituents (mainly hemicellulose fragments) increase the liquor viscosity [4, 5] and silica may cause scaling problems in the evaporation plant. During the past decade, techniques for avoiding both polysaccharide and silica problems have been developed. For example, several alternative methods have been proposed to remove silica from black liquor by precipitation or by making silica more soluble. One of the

most attractive precipitation processes is to lower the liquor pH to about 10 by carbon dioxide or in practical applications by carbon dioxide from flue gases [6]. In addition, it is possible to hold silica in the dissolved form in the liquid phase by adjusting residual alkali of the black liquor to a level corresponding to a pH greater than 12 [7]. The liquor heat treatment system used in the wood-utilizing pulp mills can also be applied to nonwood-utilizing mills to reduce the viscosity of black liquor, mainly by decreasing the polysaccharide content of black liquor [7].

Based on the present trends in nonwood pulping, we can conclude that understanding the burning characteristics of different nonwood black liquors is essential for the efficient burning of these liquors in the recovery furnace and for the further design of the mill's recovery unit. Combustion properties of various black liquors have been studied under controlled laboratory conditions by burning single liquor droplets or larger quantities of black liquor in a proper furnace under different gas atmospheres [8-10]. In these studies, the following burning parameters have generally been studied: duration of the burning stages (including drying, devolatilization or pyrolysis, and char burning), degree of swelling during the devolatilization stage, various conversion yields and rates, and formation rates of numerous gaseous products.

Black liquor contains, in addition to inorganic substances, polysaccharide degradation products (aliphatic carboxylic acids and hemicellulose residues), and minor amounts of extractives. Understanding of the possible relationships between the main organic constituents and the different combustion stages of a black liquor droplet is important when considering the combustion properties of various black liquors [11]. The aliphatic carboxylic acids have a significant influence on the drying rate of black liquor droplets, but also on swelling during the devolatilization stage, due to the instability of these acids when heated. In contrast, lignin has no such straightforward role in combustion, although its aromatic moieties are obviously important with respect to char formation and burning. In addition, there are indications that the high-molecular-mass lignin material, together with hemicellulose residues, plays an important role in hindering the escape of volatile products from within a black liquor droplet during the devolatilization stage.

FTIR spectroscopy is a rapid method for indicating different chemical bonds and thus different functional groups in wood and pulp samples [12-15]. For this reason, with respect to the topics related to pulping, there are several FTIR-based techniques for measuring, for example, the kappa number of pulp or the contents of lignin and carbohydrates in wood and pulp. Since there exist correlations between the content of black liquor constituents and the combustion properties of black liquor, it could be further concluded that correlations between these properties and, for example, easily detectable FTIR data, offer an interesting possibility for developing a proper method to characterize the burning behavior of black liquor.

The main purpose of this study was to determine the combustion properties of nonwood black liquors from soda-anthraquinone (AQ) pulping of wheat straw and soda-AQ, and kraft pulping of reed canary grass. The secondary aim was to explore possibilities for predicting the burning behavior of these black liquors by means of their chemical composition. In this study, we selected FTIR spectroscopy as a rapid analysis technique to obtain chemical data on black liquors.

EXPERIMENTAL

Liquor samples and heat treatments

Black liquor samples originated from laboratory pulping of wheat straw and reed canary grass (*Phalaris arundinacea* L.). Soda-AQ pulping was applied to both feedstock materials. In addition, we delignified the reed canary grass by kraft pulping. **Table I** presents the detailed cooking parameters.

Pulp yields, kappa numbers, and other pulp properties of wheat straw and reed canary grass pulps from soda-AQ pulping are presented in **Table II** and the corresponding information on reed canary grass pulps from kraft pulping in **Table III**.

Combustion experiments

Single black liquor droplets were weighed and combusted in stagnant air at 800°C in a laboratory furnace similar to that developed by Hupa and coworkers [18]. Dry solids contents of the combusted liquors were adjusted by vacuum evaporation to an approximate value of 45%; the droplet mass varied within 6-10 mg. We recorded combustion with a digital video camcorder and determined the duration of different burning stages (devolatilization and char burning) and the size of the swollen droplets. We performed 12-15 parallel determinations for each liquor to ensure reliability of the measurements. In this paper, we also illustrate the overall duration of combustion stages of black liquor for each liquor by means of a calculated variable: burning time. This term expresses the sum of the burning times determined separately for the devolatilization and char burning stages.

FTIR determinations

The FTIR measurements were performed in each case from a thin pellet prepared from a homogenized mixture of oven-dried black liquor and potassium bromide (KBr, about 200 mg, mass proportion 1:40, respectively). The analysis was carried out with a Nicolet Magna-IR 550 Series II FTIR spectrometer in the wave number range 400–4000 cm^{-1} . In each measurement, 128 scans were accumulated at 4 cm^{-1} resolution. To obtain the background contribution, the KBr spectrum was measured for automatic background subtraction. In addition, we eliminated the absorbance peaks of water (i.e., air humidity) and CO_2 by subtracting these peaks from the spectra of the sample mixtures.

RESULTS

Combustion properties

The influence of cooking parameters (cooking temperature and time) and heat treatment on the characteristic burning times and swelling for wheat straw black liquors from soda-AQ pulping are given in **Figs. 1** and **2**, respectively. As can be seen from Fig. 1, for untreated liquors obtained at 0 min cooking time, the burning times of liquor droplets were almost equal and generally due to shorter char burning times. The burning times were also slightly shorter than those obtained at 30 min cooking time. In addition, these values were at the same level as detected earlier for liquor droplets from conventional birch kraft pulping [19].

Due to the multitude of possible reactions taking place during heat treatment, no straightforward explanation could be given for a general trend that the burning times were longer for the untreated liquor droplets than those for the heat-treated liquor droplets. However, this trend can be partly explained by assuming that high-molecular-mass hemicellulose and lignin residues in black liquor are degraded to some extent to lower-molecular-mass fragments on heating. For this reason, data on the chemical composition of the black liquors studied would be of great importance and detailed analyses of these liquors will be carried out separately in a forthcoming investigation.

The wheat straw black liquors from soda-AQ pulping swelled slightly more during the devolatilization stage than birch black liquors from conventional kraft pulping (Fig. 2) [19]. As cooking time increased, the droplets also swelled more during this stage. This finding agreed well with earlier data on wood black liquors [19]. On the other hand, no significant differences in swelling were evident between the untreated and heat-treated liquor droplets in these cases.

The results indicated that the char burning time of the untreated reed canary grass black liquors from soda-AQ pulping steadily decreased as cooking temperature increased (**Fig. 3**). In contrast, the same trend was not seen for the heat-treated liquors, which generally had shorter burning times than those detected for the untreated liquors. As a general trend, the heat-treated liquor droplets showed a higher degree of swelling than the untreated ones (**Fig. 4**).

In the case of reed canary grass black liquors from kraft pulping, variations in the sulfidity used in delignification caused the most obvious differences between the combustion properties of different black liquors (**Figs. 5 and 6**). In practice this meant, for example, that an accelerated dissolution and degradation of lignin occurred at higher sulfidity and the burning times were significantly longer for liquors formed with 38% sulfidity than for those with 15% sulfidity. In addition, the latter liquor droplets (sulfidity 15%) swelled clearly more than the former ones (sulfidity 38%). Surprisingly, we noticed a similar burning behavior for the untreated and treated black liquors at the same sulfidity level.

In the case of reed canary grass black liquors, then, we found the longest burning times and the least swelling for kraft black liquors when compared to the corresponding soda-AQ black liquors (**Fig. 7**). Understandably, due to their large surface area, the more swelled droplets were also more amenable to various reactions and burned faster than the less swelled droplets. We obtained an even a better correlation ($R^2 = 0.68$) between swelling and char burning time, but surprisingly, found practically no correlation ($R^2 = 0.01$) between swelling and devolatilization time. It is evident that the latter finding seems somewhat conflicting since swelling takes place during the devolatilization stage and the maximum size of the droplet occurs at the end of this stage. However, we could not find any correlation between swelling and burning time or char burning time for wheat straw black liquors (**Fig. 8**).

FTIR correlation tests

FTIR spectroscopy offers one of the potential methods to rapidly detect differences in the chemical composition of various black liquors. However, due to the complex nature of black liquor dry solids, the straightforward interpretation of the spectral data on black liquors was not easy. In addition, in FTIR spectroscopy one chemical component may absorb at several frequencies, while two or more components may contribute to the same absorption band. **Table IV** shows some examples of the assignments of various absorption bands (peaks 1 $\rightarrow$ 6) in the wave number region 1000-3000 cm^{-1} . These peaks originated typically from different wood constituents and were selected for this purpose. **Figure 9** gives examples of FTIR spectra of the untreated and heat-treated reed canary grass black liquors from soda-AQ pulping.

In the initial experiments, the purpose was to study whether there are distinct differences in the intensities of the selected peaks in the FTIR spectra of various black liquors. It soon became clear that adequate information can be simply obtained firstly by measuring certain FTIR peak intensity ratios for the black liquors studied and secondly by plotting the same ratios against the typical parameters characterizing the burning behavior of these black liquors. In this study, we tested only relatively simple ratios, where the denominator used was the intensity of peak 3 (Table IV), typical for lignin. However, it is also possible to form a great number of other ratios based on the proper peaks. In addition, multivariate analyses were not used to handle the FTIR spectral data in this instance; this kind of approach will be carried out separately.

Figures 10-13 give examples of the FTIR peak intensity ratios for the black liquors studied. In most of these examples, clear differences between the untreated and heat-treated liquors were obtained. These intensity ratios are plotted against the characteristic burning times and swelling for

all black liquors in **Fig. 14**. As can be seen, with respect to the possibilities of predicting the burning behavior of individual black liquors, most of the correlations shown are useful. However, if needed, the wide choice of FTIR peaks greatly facilitates the selection of other appropriate peak intensity ratios.

CONCLUSIONS

Wheat straw and reed canary grass black liquors obtained from laboratory soda-AQ and kraft pulping all showed similar burning behavior, which is also typical for the corresponding birch black liquors. In case of kraft pulping, the sulfidity used in delignification had a significant influence on the swelling properties of the formed black liquors. The heat treatment, which is necessary for nonwood black liquors to reduce their viscosity prior to evaporation resulted in a slight decrease in burning time in the case of soda-AQ liquors, but had practically no influence on swelling. In addition, the results indicated that the primary combustion properties could be predicted by means of FTIR spectral data. Although this method seemed to have potential for characterizing the burning behavior of different nonwood black liquors, further work is needed, especially in the detailed chemical analysis of these kinds of black liquors.

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I. Pulping data on soda-AQ pulping of wheat straw and reed canary grass and on kraft pulping of reed canary grass.

Parameter	Unit	Soda-AQ	Kraft
Effective alkali as NaOH	% on o.d. feedstock	18	18
AQ charge	% on o.d. feedstock	0.05	0
Sulfidity as NaOH	%	-	15, 38
Maximum temperature	°C	145, 155, 165	165
Time at maximum temperature	min	0, 30	0, 30
Time from 25°C to max. temp.	min	90	90

Liquor-to-feedstock ratio was 6:1 L/kg o.d. feedstock in wheat straw pulping and 5:1 L/kg o.d. feedstock in reed canary grass pulping.

II. Pulp properties of wheat straw and reed canary grass pulps from soda-AQ pulping [16, 17].

Max. temp.	Time (min)	Reed canary grass				Wheat straw			
		Screened yield (%)	Rejects (%)	Kappa number	Viscosity (mL/g)	Screened yield (%)	Rejects (%)	Kappa number	Viscosity (mL/g)
145	0	44.9	4.7	28.7	1105	35.5	8.8	20.2	993
145	30	43.5	3.9	17.9	1210	38.8	4.7	12.6	1083
155	0	44.5	4.7	24.1	1131	35.0	8.4	17.7	1040
155	30	44.0	2.3	14.0	1251	40.3	1.7	9.5	1155
165	0	43.9	2.9	18.5	1201	38.3	3.4	13.3	1101
165	30	43.8	1.2	12.3	1266	40.9	1.0	8.2	1157

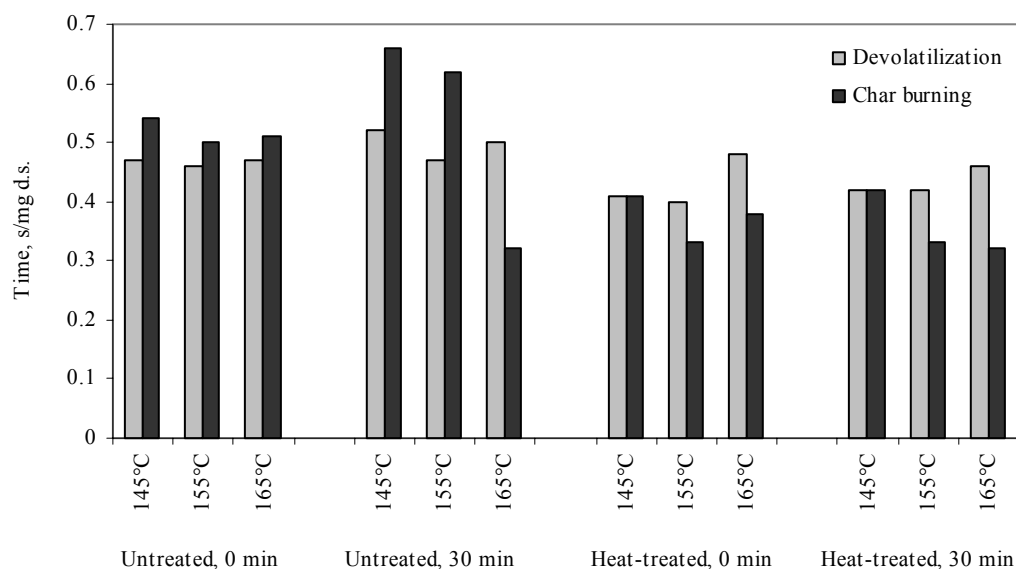
III. Pulp properties of reed canary grass pulps from kraft pulping.

Sulfidity (% as NaOH)	Time at max. temp. (min)	Screened yield (%)	Rejects (%)	Kappa number	Viscosity (mL/g)
15	0	47.2	2.8	20.5	1306
15	30	47.0	0.7	14.6	1297
38	0	48.0	2.0	16.3	1342
38	30	50.3	0.1	12.9	1232

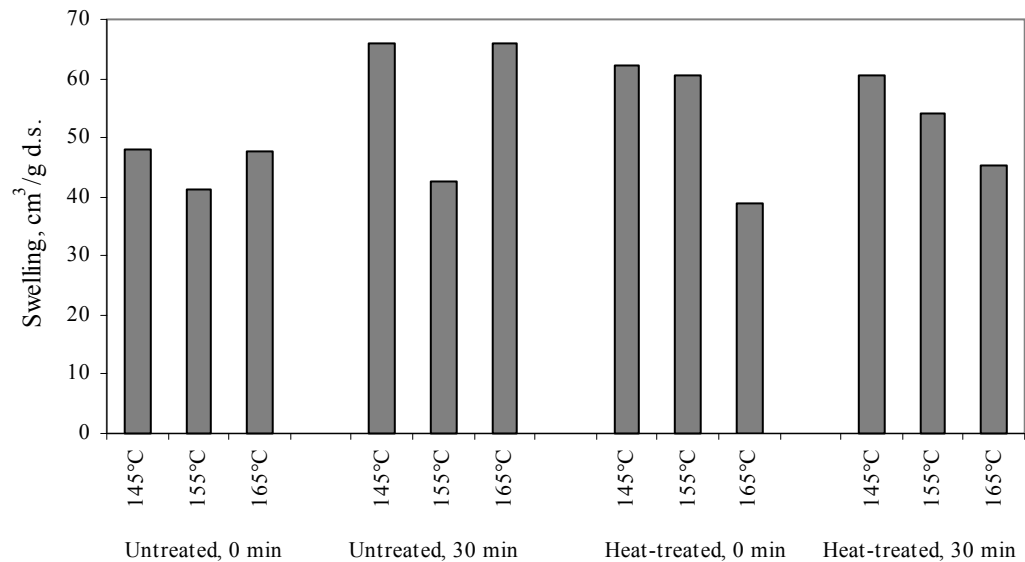
Heat treatments of black liquors were carried out by keeping each sample in a nitrogen-purged reactor in an oil bath at 190°C for 30 min.

IV. Assignment of the infrared absorption bands in the spectra of wheat straw and reed canary grass black liquors.

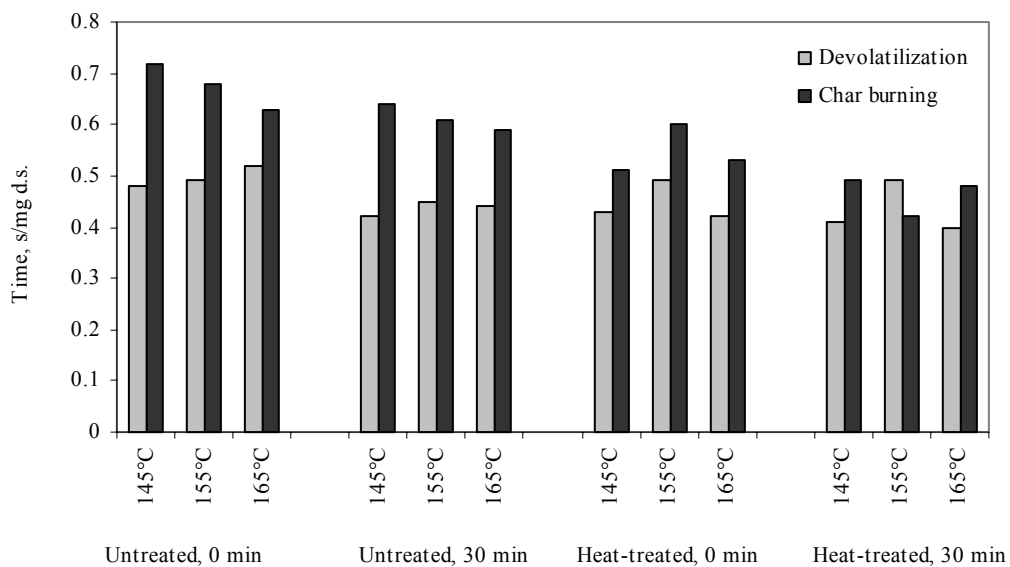
Peak	Wavenumber, cm ⁻¹	Source	Assignment	Ref.
1	2930-2940	cellulose, hemicellulose lignin	CH ₂ antisymm. stretching O-H stretch. in methyl and methylene groups	20,21 22
2	1595-1605	lignin	aromatic ring C=C vibr. and C=O stretch.	22-24
3	1500-1510	lignin	aromatic ring C=C vibr.	22,24,25
4	1415-1425	hemicellulose cellulose lignin	C-H stretch. of CH ₃ and OCH ₃ groups CH ₂ bending aromatic ring vibr. and C-H deform.	26 27,28 22,23,25
5	1110-1120	cellulose, hemicellulose cellulose lignin	O-H assoc. C-O and C-C stretch. and CH ₂ rocking Ring breathing and C-O stretch.	29 28 15
6	1040-1050	cellulose lignin and hemicellulose (LCC*) glucuronoxylan lignin	C-O stretch. C-O stretch. C-O stretch. alkyl C-O vibr.	27 29 15 30



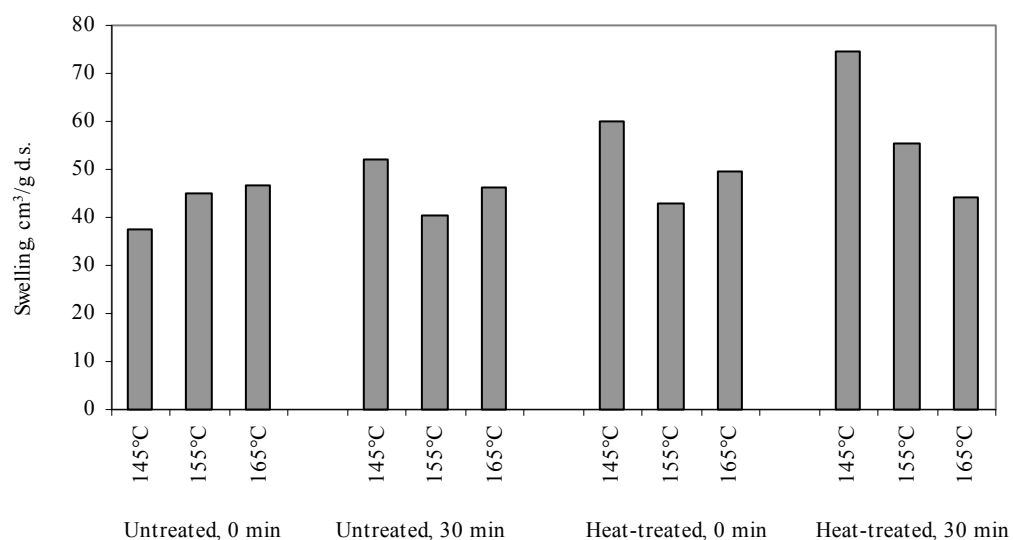
1. The influence of cooking parameters (cooking temperature and time) and heat treatment on devolatilization and char burning times in case of wheat straw black liquors from soda-AQ pulping.



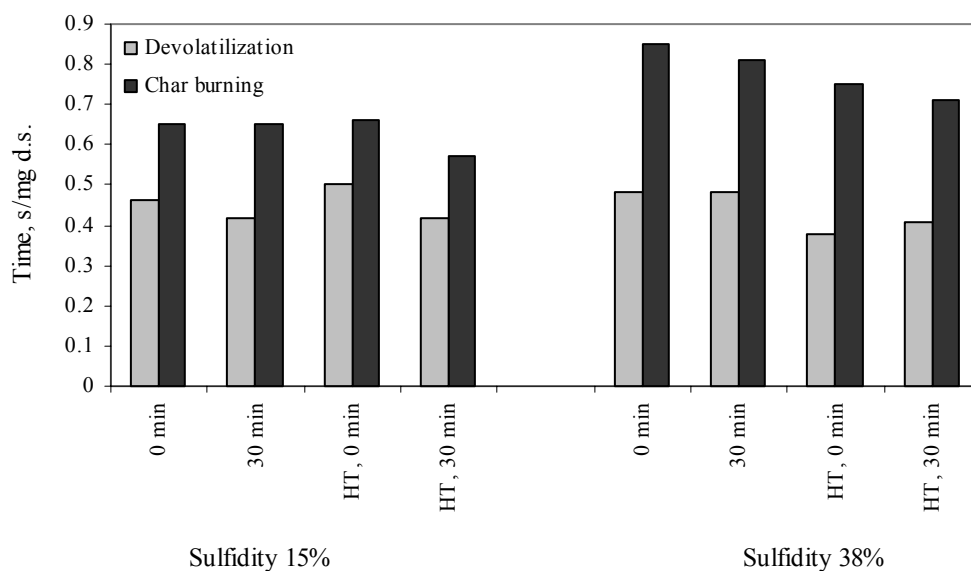
2. The influence of cooking parameters (cooking temperature and time) and heat treatment on swelling in case of wheat straw black liquors from soda-AQ pulping.



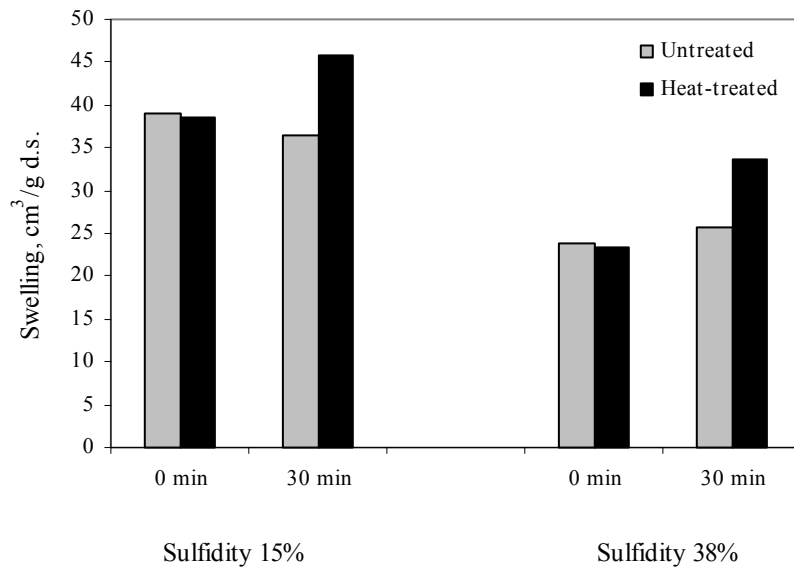
3. The influence of cooking parameters (cooking temperature and time) and heat treatment on devolatilization and char burning times in case of reed canary grass black liquors from soda-AQ pulping.



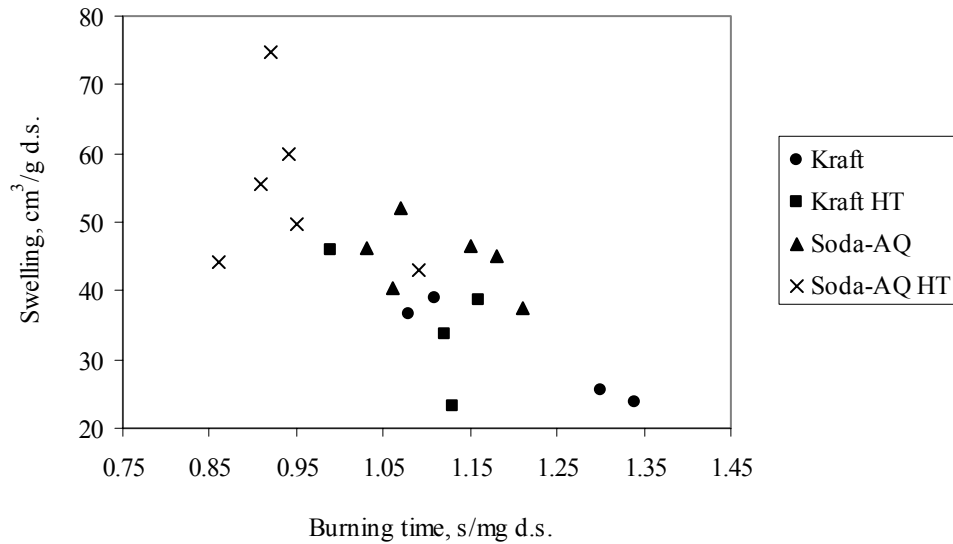
4. The influence of cooking parameters (cooking temperature and time) and heat treatment on swelling in case of reed canary grass black liquors from soda-AQ pulping.



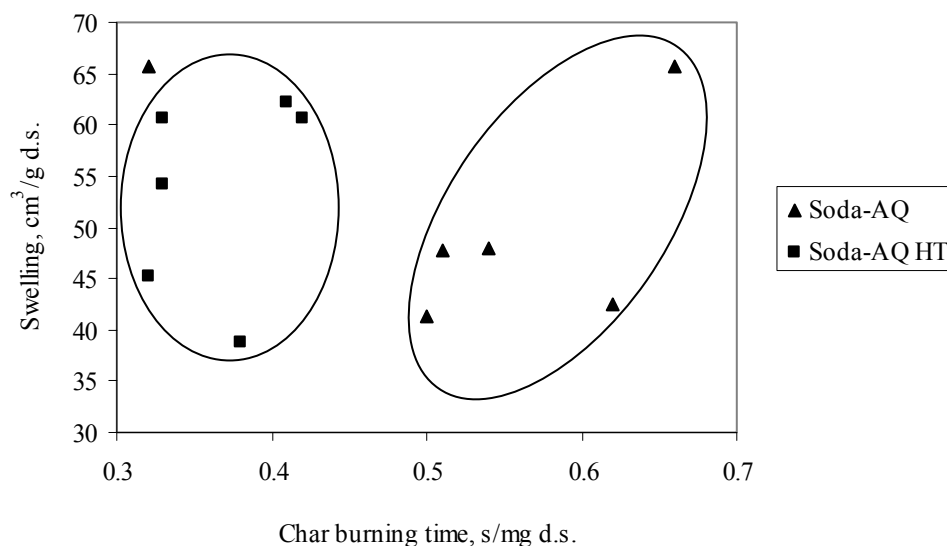
5. Devolatilization and char burning times of the untreated and heat-treated (HT) reed canary grass black liquors from kraft pulping with different sulfidities and cooking times at maximum temperature 165°C.



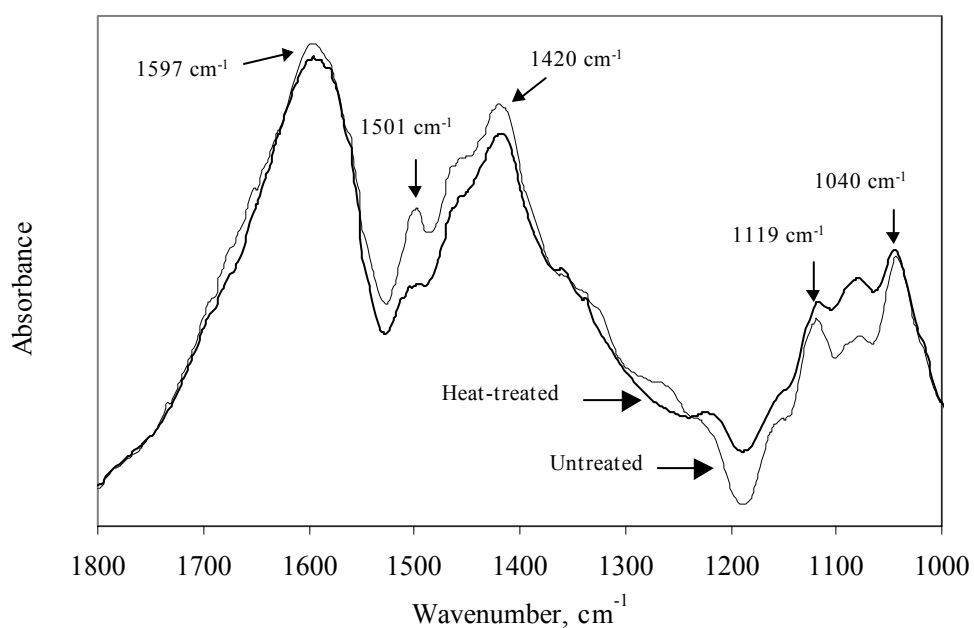
6. Swelling of the untreated and heat-treated reed canary grass black liquors from kraft pulping with different sulfidities and cooking times at maximum temperature 165°C.



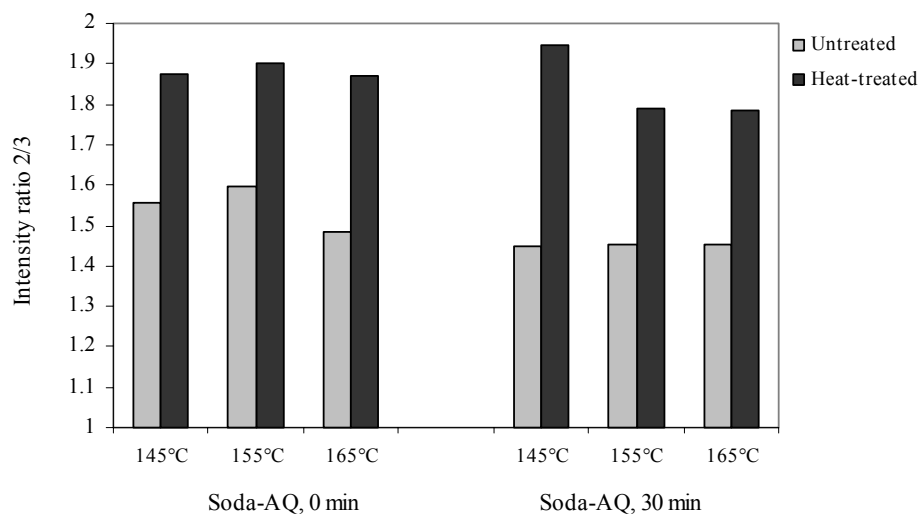
7. Swelling vs. burning time in cases of the untreated and heat-treated (HT) reed canary grass black liquors from soda-AQ and kraft pulping ($R^2 = 0.55$).



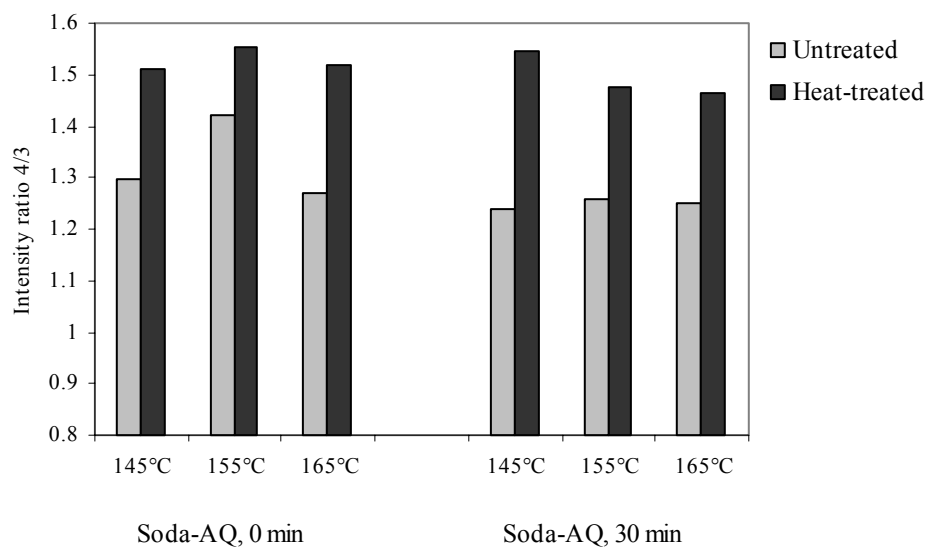
8. Swelling vs. char burning time in case of the untreated and heat-treated (HT) wheat straw black liquors from soda-AQ pulping.



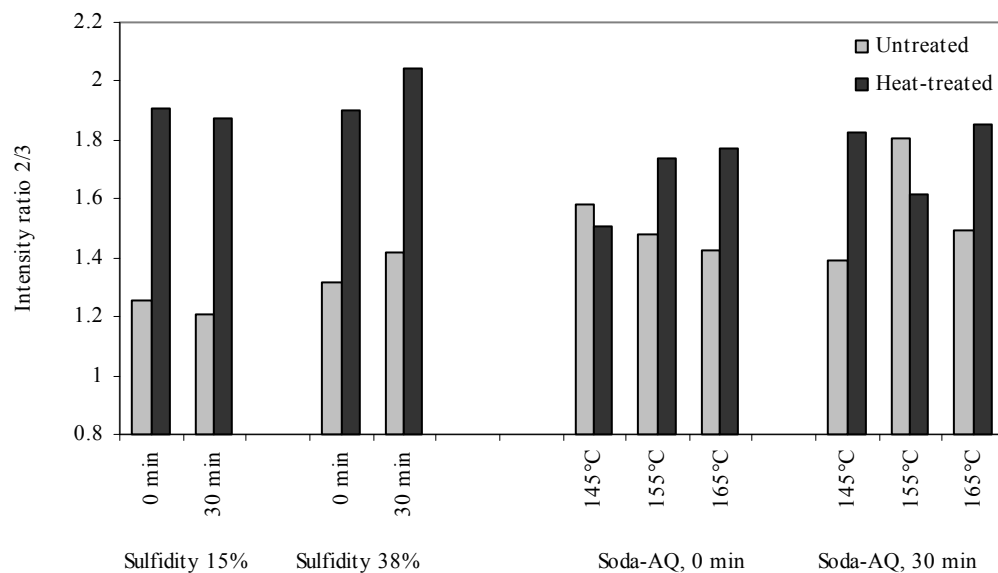
9. Example of FTIR spectra of the untreated and the heat-treated reed canary grass black liquor from soda-AQ pulping. Cooking temperature was 165°C and cooking time at maximum temperature 30 min.



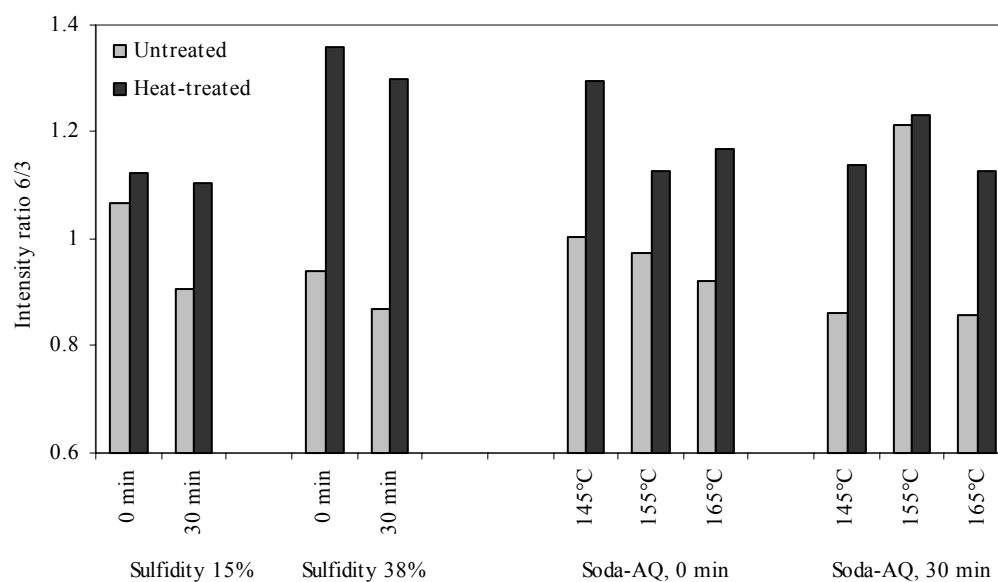
10. FTIR peak intensity ratio of 2:3 for the untreated and heat-treated wheat straw black liquors from soda-AQ pulping. For the origin of peaks 2 and 3, see Table IV.



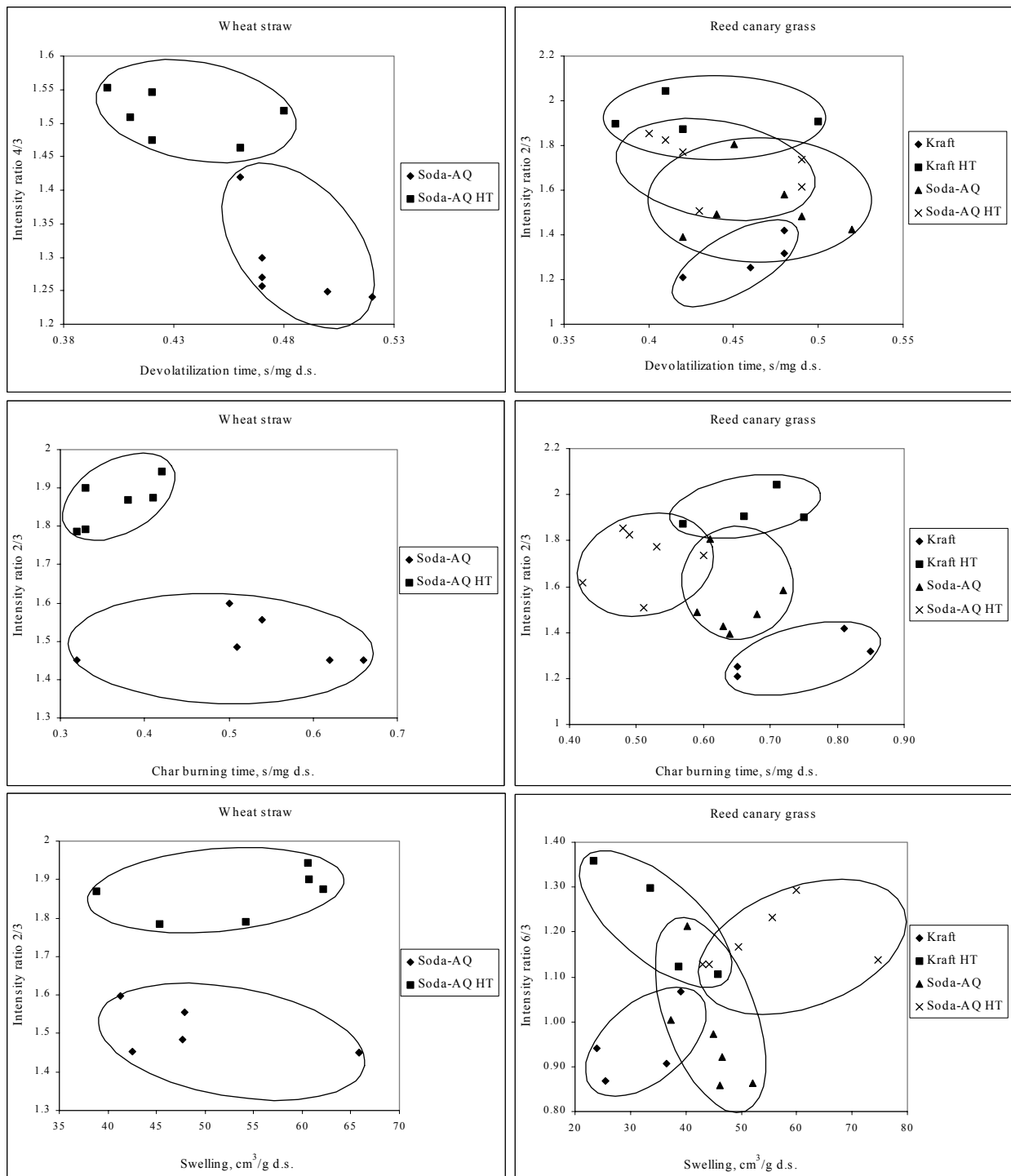
11. FTIR peak intensity ratio of 4:3 for the untreated and heat-treated wheat straw black liquors from soda-AQ pulping. For the origin of peaks 3 and 4, see Table IV.



12. FTIR peak intensity ratio of 2:3 for the untreated and heat-treated reed canary grass black liquors from kraft and soda-AQ pulping. For the origin of peaks 2 and 3, see Table IV.



13. FTIR peak intensity ratio of 6:3 for the untreated and heat-treated reed canary grass black liquors from kraft and soda-AQ pulping. For the origin of peaks 3 and 6, see Table IV.



14. FTIR peak intensity ratios vs. combustion parameters in case of the black liquors studied.

Author Insights

Combustion in the recovery furnace offers, at present, the most applicable way to recover cooking chemicals and energy from nonwood black liquors. By heat pretreating these liquors, we can evaporate them to higher dry solids content than is possible conventionally, thus enhancing the energy production.

Although the combustion properties of wood black liquors have been studied extensively, little comprehensive information is available on the combustion properties of nonwood black liquors.

In this study, it was interesting to notice that combustion properties of nonwood black liquors did not differ much from those of hardwood black liquors. The most interesting scientific finding was probably the detection of systematic changes in the FTIR spectra of black liquors taking place during heating.

FTIR data can be used, for example, to predict the combustion properties of heat-treated black liquors. The next step of this research will be to obtain detailed information about the chemical composition of nonwood black liquors and consequently to find out possible correlations between composition and combustion properties data. In addition, we will take a closer look at whether the data on chemical composition explain the detected changes in the FTIR data. This kind of information would be useful when developing control methods for black liquor recovery process, for example.

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