Gasification of mixtures of black liquor and secondary sludge

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ABSTRACT: The effects of secondary sludge addition on black liquor gasification were studied in a laminar entrained flow reactor. Additions of 0%, 5%, 10%, and 15% secondary sludge solids to hardwood black liquor solids were studied at gasification temperatures of 700°C and 900°C with residence times of 0.2, 0.7, and 1.2 s. Although both the amount and the rate of organic carbon conversion increased with temperature and residence time, the percentage of sludge addition did not affect the percentage of feed carbon recovered as organic carbon. Recoveries were expressed on a percentage-of-feed-element basis. Recoveries of H_2S were higher for purer (less sludge added) feed stocks for all experiments except those performed at 900°C and 1.2 s residence time. Recoveries of methyl mercaptan, n-propyl mercaptan, carbonyl sulfide, and ammonia were relatively independent of percentage sludge addition. Amount of ammonia recovered, however, increased at higher sludge addition rates.

Application: With the development of black liquor gasification nearing commercialization, this study lays the groundwork for combining black liquor and secondary sludge in a process which provides an energy-efficient method for sludge disposal.

Secondary sludge is the settled residue that accumulates in a secondary clarifier during the wastewater treatment process. Currently, landfilling is the most popular disposal method for secondary sludge, but tipping fees, hauling costs, and special landfilling requirements for hazardous sludges are major drawbacks to this option. Alternative sludge disposal methods include burning in recovery boilers, land application, incineration, and lagooning. Although land-applied sludge nourishes vegetation, conditions soil, and helps break down pesticides, disadvantages do exist. Hauling costs, limited land availability, dilution costs, odor problems, and groundwater leaching are a few of the problems associated with land application. Incineration significantly reduces the amount of material to be landfilled and recovers a portion of the energy from the combusted biomass; however, this process produces a flue gas stream containing NO_x, SO_x, and organic halide compounds that must be captured and treated if produced in quantities that exceed permit levels. A potential sludge disposal method not currently being utilized is sludge addition to a black liquor gasification unit [1].

Gasification of black liquor is under active development for use either as a replacement for kraft recovery boilers or for incremental black liquor recovery. Gasification is the process of producing a combustible fuel gas from black liquor, which can be combusted in an integrated combined cycle scheme to drive a gas turbine and produce electricity. One unique advantage of black liquor gasification over traditional combustion is the ability to separate out sulfur and sodium. Depending on gasification temperature, a large fraction of the sulfur can be volatilized and exit with the fuel gas. This fuel gas sulfur is then scrubbed and can be used to produce polysulfide pulping liquors or kraft liquors with varying sulfidity. This provides an opportunity to utilize more advanced pulping methods [2].

The goal of this study was to examine the feasibility of gasifying black liquor mixed with kraft mill secondary sludge. Before this can be done with confidence, a number of technical issues brought about by differences in composition between black liquor and secondary sludge must be considered. In a typical kraft mill, around 58 kg of dewatered sludge are produced per metric ton of pulp [1]. Sulfite mills produce sludge on the order of 102 kg/metric ton of pulp, while deinking mill sludge production is around 234 kg/metric ton of pulp. While typical black liquor has a heating value of around 13.4 to 15.5 MJ/kg, **Table I** indicates significantly higher values for kraft paper mill sludges, making them as efficient an energy source as bark or wood chips.

Sludge from mill wastewater contains virtually all of the compounds introduced to the process streams by pulping/bleaching reactions, process additives, and equipment corrosion. Inert nonprocess element (NPE) effluent compounds that arise from the closure of mill processes tend to accumulate in pulp and paper mill process loops and

		ANALYSIS (%)					HEATING VALUE
Source	Solids	Ash	C	H	S	O	N	(MJ/kg)
Bleached pulp mill	33.4	1.9	48.7	6.6	0.2	42.4	0.2	20.1
Typical pulp mill	42.0	4.9	51.6	5.7	0.9	29.3	0.9	21.5
Kraft mill	37.6	7.1	55.2	6.4	1.0	26.0	4.4	24.1
Kraft mill (2nd source)	40.0	8.0	48.0	5.7	0.8	36.3	1.2	19.8
Deinking mill	42.0	20.2	28.8	3.5	0.2	18.8	0.5	12.0
Deinking mill (2nd source)	42.0	14.0	31.1	4.4	0.2	30.1	0.9	12.2
Recycle mill	45.0	3.0	48.4	6.6	0.2	41.3	0.5	20.8
Bark alone	54.0	3.5	48.0	6.0	0.1	42.1	0.3	20.3
Bark alone (2nd source)	50.0	0.4	50.3	6.2	0.0	43.1	0.0	20.8
Wood chips	79.5	0.2	49.2	6.7	0.2	43.6	0.1	19.4

I. Elemental compositions and heating values of various sludges, bark, and wood chips [1].

	ELEMENT	AVERAGE	RANGE
Macro-wood elements (%)			
	N	2.9	0.14-4.1
	P	0.34	0.125-0.58
	K	0.137	0.037-0.595
	Ca	2.95	0.266-9.02
	Mg	0.065	0.011-0.100
Micro-wood elements (ppm)			
	Fe	2,930	675-7162
	B	13.3	8.5-21.1
	Cu	67.4	2.2-90.9
	Zn	127.0	41.3-213.6
	Mn	119.0	22.9-286.4
	Mo	6.7	5.2-8.9
Other elements (ppm)			
	Na	1730	490-4120
	Al	19,500	15,000-22,000
	Cd	3.9	1.6-9.4
	Cr	39.6	29.2-55.8
	Pb	81.9	37.8-129.3
	Hg	3.9	3.9

II. Composition of various combined pulp and paper mill sludges on an ovendry basis [3].

slowly exit with sludge over time. In addition to organics, sludge contains inorganic ash, including toxic sludge components such as cadmium, chromium, and heavy metals in parts-per-million (ppm) concentrations. **Table II** summarizes the elemental compositions of a variety of pulp and paper mill sludges.

About twice as much nitrogen appears in pulp mill sludge as enters with wood. This is due primarily to the addition of nitrogen to the effluent treatment process as a bacterial nutrient. According to [3], nitrogen makes up about 2.9% of combined sludge. Since nitrogen, on average, constitutes only about 0.13% of black liquor, there is concern that gasifying black liquor mixed with sludge could produce elevated NO_x levels compared to those produced from gasification of black liquor alone [2]. Four methods are responsible for forming NO_x during gasification and combustion: nitrogen release, formation from char, formation from volatile nitrogen, and formation from nitrogen in combustion air. Devolatilization products are molecular nitrogen, ammonia, and char nitrogen [4, 5]. Temperature increases enhance the release of nitrogen, even during post-volatilization time periods. After devolatilization, NO_x can form from oxidized char. NO_x can also be formed from volatile nitrogen when ammonia, the main reactive nitrogen intermediate released during pyrolysis, is oxidized. Ammonia can also react with NO in the presence of oxygen to form more NO [5]. Ammonia conversion to N_2 is also possible, according to [4]. Interestingly, another study has shown that recovery boiler smelt can decompose NO into N_2 and O_2 [6].

If phosphorus-containing detergents are used in the pulping process, the amount of phosphorus exiting the mill with sludge can be more than twice the amount entering with wood. Typically, about one-quarter of the potassium entering the pulping process with wood exits as a component of sludge. Even though calcium is the most abundant mineral found in wood, about twice as much calcium exits pulp mills with sludge than enters with wood. This is due to the fact that very large quantities of calcium are added in the pulping and bleaching processes. Though iron is present at low levels in wood, its concentration in pulp mill sludge is fairly high due to ions introduced through equipment

Input of nonprocess elements (kg/air-dried metric ton)

Source	Al	Ca	Cl	Mg	P	Si
Mill I sludge	0.23	0.11	0.03	0.08	0.07	0.10
Mill II sludge	0.33	0.20	0.005	0.12	0.24	0.23
Mill III sludge	0.39	0.09	0.005	0.14	0.18	0.59
Mill IV sludge	1.73	1.78	0.07	0.16	0	1.13
Wood and make-up chemicals[a]	0.06	2.46[b]	1.08	0.53	0.2[c]	1.26

[a] from [7] (except for phosphorus)
[b] excluding make-up lime
[c] estimated from phosphorus content of wood [8]

III. Input rates of nonprocess elements to a kraft recovery system [9].

corrosion. Cadmium is a plant toxin and is found in trace amounts in pulp chips; however, it can enter the pulp mill with detergents, pigments, and plastic/PVC tubing. Because of its tendency to concentrate in sludge, cadmium levels have been regulated in sludge used in agricultural applications. While copper ions never reach appreciable levels in sludge, aluminum is present in paper mill effluents in very large amounts from use of clay coatings and alum. Although a large quantity of sodium is absorbed by the pulp, about twice as much sodium leaves with sludge as enters with wood [3].

Inorganic nonprocess elements will inevitably be added to the recovery cycle upon introduction of sludge to a gasifier. **Table III** details these additions for sludge incineration.

Adding sludge to an incinerator at a rate of 15 kg per air-dried metric ton of pulp increases aluminum content by about 10 times; calcium, magnesium, phosphorus, and silica levels increase by 10% to 100% and chlorine by less than 10%. Since chlorine and aluminum are soluble in kraft process liquors, these elements will tend to build up within the recovery cycle. Accumulation of aluminum could cause evaporator fouling, while accumulation of chlorine can accelerate corrosion. Silica is moderately difficult to remove from the recovery process, but may in part be removed through dregs and grits purges. Residual silica can build up in the lime cycle, where a lime mud purge may become necessary [9]. Adding sludge directly to a black liquor evaporator can reduce the heat transfer coefficient by leaving a slimy deposit on evaporator tubes [9]. Most NPEs are effectively purged in the green liquor clarifier underflow, which goes to the dregs filter. However, elevated dregs levels in clarified green liquor can result in calcium carryover in the white liquor, decreased lime mud settling rate in the white liquor clarifier, decreased lime mud washing efficiency, poor weak-wash clarity, increased TRS lime kiln emissions, and increased fuel requirements in the lime cycle [10].

Because of the differences in physical and chemical properties between black liquor and secondary sludge, determination of the feasibility of gasifying mixtures of these streams requires that the following issues be addressed:

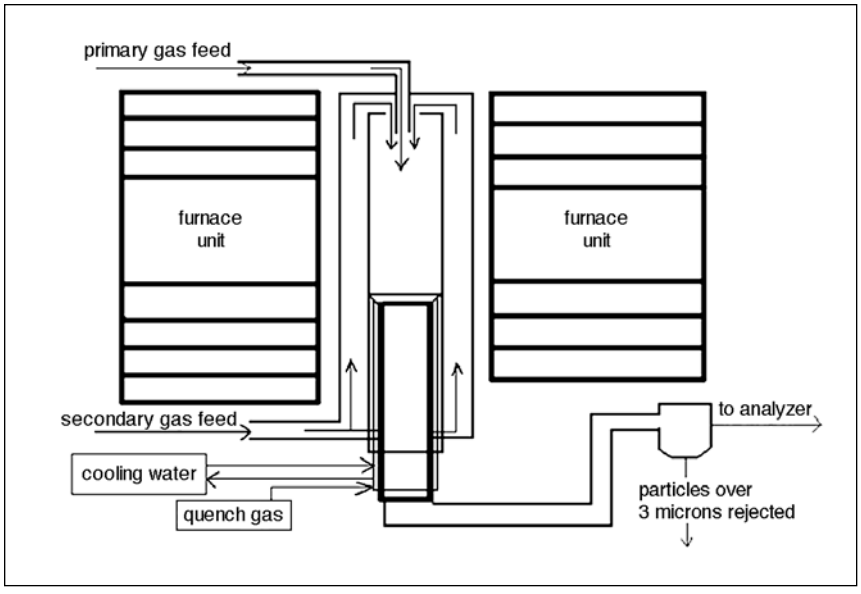
1. Although secondary sludge has a relatively high organic content, it may reduce the heating value of the fuel gas obtained from gasification and may render the black liquor fuel gas stream a less efficient source of electricity.
2. The char from the gasification of secondary sludge may have a negative effect on overall gasification rates. Rate data for gasification of mixtures of sludge and black liquor are needed for comparison with gasification of black liquor alone.
3. Because secondary sludge has high nitrogen content compared to black liquor, NO_x precursor levels produced from the gasification of the sludge/liquor mixture must be compared to those produced from gasification of black liquor alone.
4. The heavy metal content of gas and condensed-phase streams produced from the sludge/liquor gasification must be compared to heavy metal contents resulting from gasifying black liquor alone.

EXPERIMENTAL

Comparison of gasification of black liquor alone versus gasification of black liquor mixed with secondary sludge must focus on gasification rate, fuel gas composition, fixed nitrogen and sulfur species content of the product gas stream, and organic carbon recovery and nonprocess element content of the product char. Independent experimental parameters investigated were gasification time, temperature, and sludge/liquor solids ratio. The sources and compositions of kraft mill sludge and black liquor were not varied.

The apparatus used for the gasification experiments (see **Fig. 1**) was a laminar entrained-flow reactor (LEFR) capable of operating at temperatures comparable to industrial gasifiers and achieving a maximum temperature of 1150°C [11]. Changing either primary/secondary flow rates or collector position changed the reactor residence time. The unit was equipped with a Fourier-transform infrared spectrometer (FTIR), a chemiluminescence NO-NO_x analyzer, and infrared CO and CO_2 analyzers. Gas chromatography was used to analyze hydrogen sulfide and ammonia concentrations in the gas stream.

Black liquor from a northeastern U.S. mill was mixed with secondary sludge from a southeastern U.S. kraft mill to the proper solids ratio (see **Table IV** for elemental analysis of sludge solids), dried, ground in a jar mill, and sieved to a particle size range of 53-125 m. Dried liquor solids entrained in the primary flow were fed into the reactor at an average rate of



1. Schematic of the LEFR [11].

0.263 g/min. Run duration was from 5 to 10 min. to ensure that steady state was achieved. Primary gas flow was maintained at about 150 mL/min, while secondary gas flow was typically about 12 L/min. Bubbling the secondary flow through a heated water bath (40.6°C) allowed water vapor to enter with the secondary gas stream, resulting in a 15/15/70 concentration of $H_2O/CO_2/N_2$ in the feed gas.

The following factorial design was completed: two gasification temperatures (700°C, 900°C), three residence times (0.2, 0.7, and 1.2 s), four black liquor/sludge ratios in the feed (100/0, 95/5, 90/10, 85/15), and two replications, giving a total of 48 runs. Most of the fumes were collected on the exhaust end of the cyclone on a 90 mm diameter filter using nylon filter paper with a pore size of 1.2 m. The balance of the fumes was collected on a 47 mm diameter filter that prevented particulate matter from exiting the cyclone and compromising the chemiluminescence NO_x meter. Because no analysis was performed on the fume samples, they were mixed with their respective char samples after masses were obtained for both. Samples from the collected mixed char and fumes were used to determine carbonate levels in the solid product using a headspace chromatography technique developed in [12]. Total carbon was determined using a high-temperature combustion/coulometric titration technique. Analyses of the reaction products included the following:

- elemental analysis of char phase for amount of nitrogen and total carbon;
- ABC titrations for carbonate in char (total carbon – carbonate = organic carbon in char);
- gas chromatography of gas phase for CO, CO_2, CH_4;
- ammonia (NO_x precursor) concentration in gas phase by chemiluminescence;
- sulfur content in gas phase with gas chromatograph;
- heavy metal (NPE) content in char using ICP (inductively-coupled plasma) and AA (atomic absorption spectrometry).

Element	Secondary Sludge, wt%
C	38.57
H	4.57
N	2.72
O*	48.99
Na	0
K	2.98
S	0.82
Other	1.35

*by difference

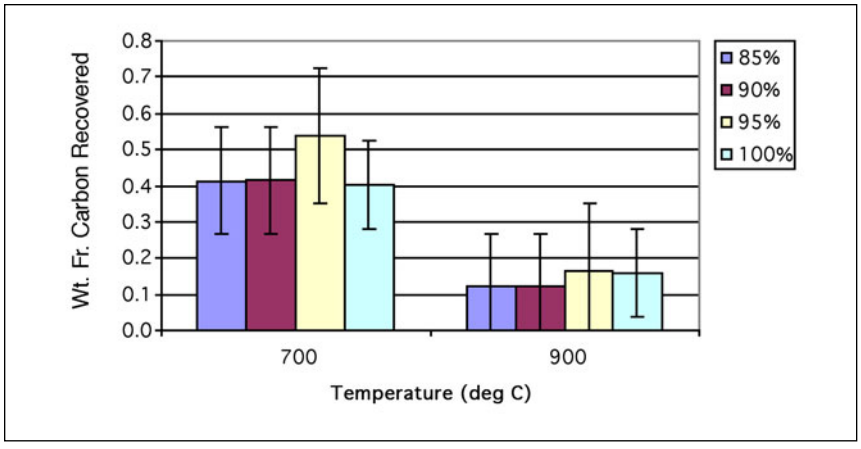
IV. Elemental analysis of secondary sludge.

Gas samples were collected with a valved Tedlar® bag once per experiment after the reaction time had reached two minutes. The following sulfur compounds were analyzed for content in the experimental product gases: COS, H_2S, methyl mercaptan, n-propyl mercaptan, ethyl mercaptan, CS_2, and isobutyl mercaptan. Because the latter three were present in levels small enough to be disregarded, only COS, H_2S, methyl mercaptan, and n-propyl mercaptan results are reported. Measurement of dimethyl sulfide was not possible because of insufficient availability of a calibration gas. (Information concerning dimethyl sulfide generation from LEFR gasification can be found in [13].) Gas samples were analyzed using an HP 5890 Series II gas chromatograph and HP Chemstation® software. A 30-m GS-GasPro® capillary column from J&W Scientific was used for sulfur gas analysis. Solids feed rates were determined from weights of the solids feed samples taken before and after each run and the reaction times. Char and fume samples were weighed after each experiment so that a percent solids recovery could be calculated. Because of cost, only the 100/0 and 85/15 feed stocks were measured for total sulfur; total sulfur values for the 95/5 and 90/10 feed stocks were calculated by interpolation.

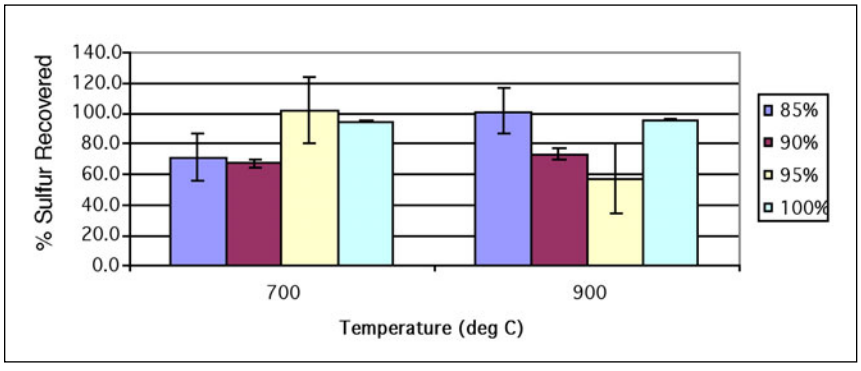
RESULTS

Product gas heating value

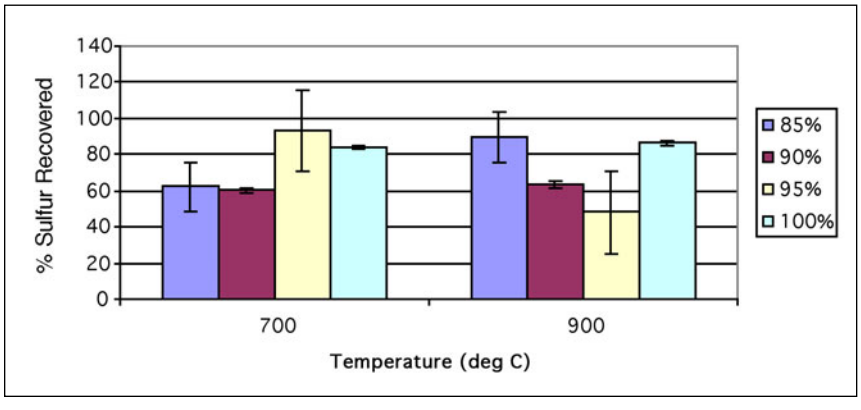
The FTIR for measuring CO and CH_4 in the product gas was unavailable, and gas chromatography proved to be ineffective because the large nitrogen peak that was generated literally enveloped the CO,



2. Effect of temperature on weight fraction carbon recovered as organic carbon at 1.2 s residence time.



3. Effect of temperature on percent feed sulfur recovered as TRS at 1.2 s residence time.



4. Effect of temperature on percent feed sulfur recovered as H_2S at 1.2 s residence time.

CH_4, and H_2 peaks. Consequently, an equilibrium simulation using HSC Equilibrium Calculation software was carried out at 900°C in an attempt to estimate the levels of CO, CH_4, and H_2 obtained from gasifying the 100/0 and 85/15 feed mixtures. Comparison of calculated results for these gases did not match experimental data obtained by Jing et al. in [14], who reported results for mixtures of black liquor and secondary sludge. It is reasonable to expect that the time-dependent data from the LEFR would yield kinetic data that are not representative of equilibrium conditions. Hence, accurate heating values for the product gases obtained in this work could not be determined.

Gasification rate

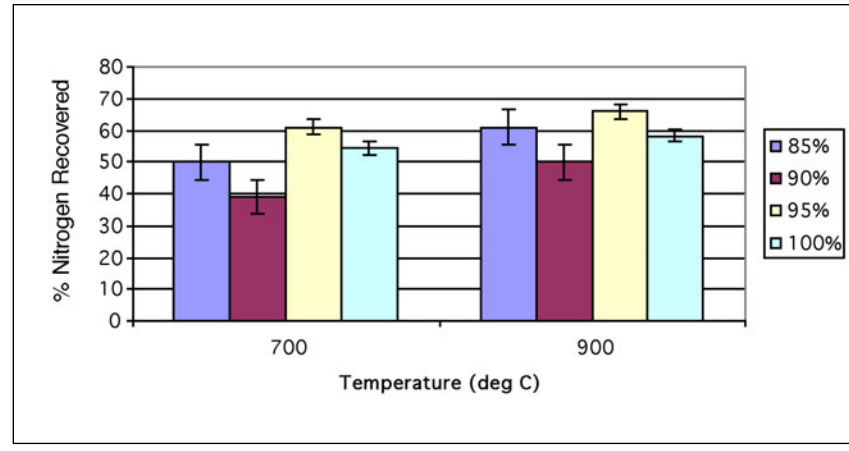
It was assumed that total carbon contained in either feed or reaction char equaled the sum of the organic carbon and carbonate carbon present. It was also assumed that there was no carbonate carbon in the feed liquors. The percentage of total feed carbon converted to inorganic salts can be used by difference to determine the degree of completion of the gasification reactions. A greater carbonate carbon recovery in the product char per mass of carbon fed to the reactor implies a greater degree of gasification. Since it was assumed that organic carbon was the only other carbonaceous material in the char, the degree of gasification completion increased in proportion to decreased recovery rates of organic carbon in the char.

Figure 2 shows the effect of temperature on organic carbon recovery at 1.2 s, with standard error bars for the four different feed materials. As expected, organic carbon recovery is reduced at 900°C, corresponding to higher conversion to carbon-containing gases, and therefore to a higher gasification rate. There do not appear to be any significant differences with feed composition at either temperature. Similar results were seen for the 0.2 s and 0.7 s residence times. Therefore, it must be concluded that while an increase in temperature from 700°C to 900°C accelerated gasification rate, up to 15% sludge addition to feed liquor had no effect on rate at a given temperature.

Gas phase sulfur content

Much data concerning sulfur gas species (H_2S, COS, MeSH, n-PrSH) in gasification product gases were obtained. Percentage of feed sulfur recovered as TRS (sum of the four listed component gases) at

1.2 s is shown in **Fig. 3**. Black liquor alone shows no temperature dependency, with over 95% of the feed sulfur recovered as TRS for both temperatures. The addition of secondary sludge generates variable results, having the same effect as or less effect than black liquor alone. With 10% sludge addition, the percentage of sulfur recovered as TRS is about 75±3%. At 15% sludge addition, results were mixed, with 71% recovery at 700°C and 100% at 900°C; at 5% sludge addition, the opposite effect was seen, with 100% recovery at 700°C and 57% at 900°C. The TRS results at 1.2 s were dominated by H_2S in that the same relative results for H_2S were seen (see **Fig. 4**). Results for the three minor TRS gases showed no significant trending relative to the percentage of sludge addition. The sum of those reduced sulfur gases generally ranged from 9% to 13%, with COS being the main contributor, ranging from 5% to 8%; methyl mercaptan ranged from 2% to 4%, while n-propyl mercaptan ranged from 2% to 3%.



5. Effect of temperature on percent feed nitrogen recovered as ammonia at 1.2 s residence time.

NOx precursor formation

Budget and time constraints did not allow for feed liquor samples to be analyzed for total nitrogen content. Because Schwanz's [15] sludge source was identical to the one used for this work, nitrogen contents in feed liquors were based on the elemental analyses reported in [15]. Recovery of feed nitrogen as ammonia increased about 5–10 percentage points as temperature increased from 700°C to 900°C for both 0.7 s and 1.2 s residence times. The results for 0.2 s residence time showed slightly greater temperature dependence, with an increase of about 15% of feed nitrogen recovered when temperature was changed from 700°C to 900°C. The 0.2 s residence time also showed the smallest generation of ammonia at about 23% to 35% of feed nitrogen recovered as ammonia. Ammonia generation increased until about 0.7 s and then leveled off at 0.7 s and 1.2 s at a steady 50% to 60% of feed nitrogen recovered as ammonia.

It was reported in [11] that after 0.5 s the fraction of feed nitrogen remaining unvolatilized stayed constant at around 50% at 900°C. Apparently, most of the volatile nitrogen transitioned to the gas phase relatively quickly upon entering the reactor. The percentage of feed nitrogen recovered as ammonia seemed relatively independent of feed liquor composition, as shown in **Fig. 5**. Because the nitrogen content (solids basis) of sludge is ~3% while that of black liquor is ~ 0.1%, from a parts-per-million output standpoint, the 85/15 liquor mixture, on average, produced a parts-per-million reading about 4-5 times that of the 100/0 liquor feed. Therefore, even though percent nitrogen recovered as ammonia was not notably dependent on liquor composition, actual total output was much higher for the liquors with higher sludge addition.

Recovery of nonprocess elements

In general, percent recoveries of NPEs at 700°C averaged about 80%; at 900°C, they averaged about 95%. This is contrary to expectation because a greater percentage of metals fed should be volatilized at the higher temperature. One probable explanation for this discrepancy is that a portion of the volatilized metals at 700°C condensed on the walls of the quench tube before entering the collection cyclone. In general, there was little difference between the recovery of specific metals in the chars for any of the liquor mixtures. However, the temperature anomalies make it impossible to draw any conclusions regarding the fate of the NPEs.

CONCLUSIONS

The percentage of total carbon recovered as organic carbon in the product char decreased with increased temperature and residence time, implying that the rate of gasification is greater at 900°C (~30% decrease after 1 s) than at 700°C (~10%–15% decrease after 1 s). No significant correlation could be established between gasification rate and percentage sludge addition to the feed liquor.

H_2S was the major sulfur species identified in the product fuel gases, with minor amounts of COS, methyl mercaptan, and n-propyl mercaptan also being formed. Ethyl mercaptan, isobutyl mercaptan, and carbon disulfide were not formed in detectable amounts during gasification at 700°C and 900°C. The black liquor/secondary sludge mixtures were seen to volatize sulfur equally well as or less readily than black liquor alone at both temperatures.

No correlation between ammonia recovery and sludge addition was observed.

However, the amount of ammonia produced by the 85/15 feed stock was four or five times greater than that produced by the 100/0 feed stock. Therefore, adding secondary sludge to a recovery process will produce more ammonia through gasification than will gasification of black liquor alone. Absolute recovery amounts correlated well with the results reported in the literature. **TJ**

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INSIGHTS FROM THE AUTHORS

The topic of this study was chosen because of today's keen interest in black liquor gasification and the possibility of combining it with an environmentally acceptable method of disposing of secondary sludge to facilitate mill closure. The study builds on previous research at IPST at Georgia Institute of Technology, both to determine experimentally the feasibility of combusting mixtures of black liquor and secondary sludge in the recovery boiler, and to address several specific challenges, including keeping the reactor feed tube from plugging by daily cleaning and obtaining accurate chemical analyses of the gaseous and condensed phase reaction products.

One of the most interesting and surprising results of this work was the disagreement between kinetic data and the predictions of equilibrium calculations. It is always reassuring if the two are reasonably close; however, kinetics must rule. The compatibility of the two feed liquors in the gasification process suggests that mills might consider adding secondary sludge to black liquor and combusting the mixture in the recovery boiler as a way to dispose of secondary sludge in an environmentally acceptable manner. This, of course, requires the recovery boiler to have unused capacity. To carry the gasification technology further, repeat experiments are needed to fill the many gaps in the data and the technology, including higher heating values of the feed mixtures, improved metals recovery, and better determination of fuel gas composition.

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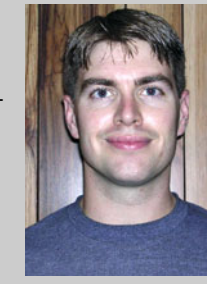

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