Energy and materials recovery from recycled paper sludge

WM. JAMES FREDERICK, KRISTIINA IISA, JAMES R. LUNDY, WILLIAM K. O'CONNOR, KAREN REIS, ANDREW T. SCOTT, SCOTT A. SINQUEFIELD, VIBOON SRICHAROENCHAIKUL, AND COLBY A. VAN VOOREN

ABSTRACT

The authors studied the pyrolysis, gasification characteristics, and the properties of the ash produced from primary sludge in a recycled fiber plant. The heating value of the sludge was 8.38 MJ/kg. The pyrolysis gases contained primarily acetaldehyde, CO, CO_2, methane, and ethylene. Evaluation of ash from a large-scale gasification test as a mineral admixture for Portland cement concrete showed it was unsatisfactory as is. Selection of better process conditions for gasification may resolve this. The authors developed a process for converting recycled fiber sludge to a low to medium heating value fuel gas with a total thermal efficiency of 70%.

THE RECYCLING OF PAPER HAS become an important industry in the past decade. Recycling reduces the requirement for virgin fiber and energy and can lower the environmental impact of paper manufacture. Over 400 U.S. facilities—more than 75% of all U.S. paper mills—now use recycled fiber. In 1993, 39.2% of the paper consumed in the United States was recovered *(1)*. The U.S. pulp and paper industry has set a 50% paper recovery goal for the year 2000 *(2)*. Current trends indicate this goal will be met *(1)*.

Over 20% of the paper recycled in the United States is coated or filled paper such as magazine stock and printing paper. This material contains a significant fraction of clay, $CaCO_3$, TiO_2, printing inks, and other additives that are not recyclable. In addition, some papermaking fibers are not reusable because of fragmentation. Recycled fiber plants processing printing grades typically generate approximately 0.5 ton of dry sludge per ton of fiber recovered. This sludge contains 40–50% water by weight. Its normal disposition is by landfill.

Because of the increasing costs of landfill disposal and the decreasing availability of sites, it is important to find alternate disposal means for the sludge from recycled paper plants. Recovery of energy, useful materials, or both from the sludge could provide an economical alternative to landfill disposal. Pilot trials have shown deinking residue can be economically treated disposed of deinking residue in a fluidized bed combustor. Air emissions were not a problem for combustion temperatures above 870°C. The fly ash produced was not a hazardous material *(3–5)*. Similar conclusions resulted from pilot gasifier trials producing a medium heating value product gas by steam gasification of sludge from a recycled paper mill *(6)*.

The disposal option considered here is converting the organic content of the sludge to fuel and reusing the resulting ash as a mineral admixture in concrete or asphalt. The study reported here characterizes sludge from a recycled fiber plant as a fuel and evaluates the residual ash as a mineral admixture for Portland cement concrete.

SLUDGE COMPOSITION AND CHARACTERISTICS

Approximately 40 L of sludge was obtained from the secondary fiber plant at the James River plant in Halsey, OR. The moisture content of the sludge as received was approximately 42% by weight (wet basis). The wet sludge had a bulk density of 600 kg/m^3, and the value for dry sludge was 354 kg/m^3. Table I provides an analysis of the sludge showing its main elemental components and heating value. The chief components were carbon, hydrogen, aluminum, silicon, calcium, and titanium. The major inorganic elements came from coatings and fillers used in the manufacture of printing papers. These included clay, calcium carbonate, and titanium dioxide. The sludge also contained approximately 0.25% by weight nitrogen; significant but minor quantities of iron, magnesium, and phosphorous; and trace quantities of other elements. The minor components shown in Table II were those present at or above the detection limit of the analytical method.

The heating value of the dry sludge solids was 8.38 MJ/kg dry solids. Its adiabatic flame temperature calculated from the heating value and composition was 1550°C.

ASH ANALYSIS

Samples of the sludge were pyrolyzed in a tube furnace at 500°C, 700°C, and 900°C for 1 h, cooled to room temperature, weighed, and analyzed for elemental composition. Table III shows that the ash residue as a percentage of dry solids content of the original sludge decreased with increasing furnace temperature from 51.5% at 500°C to approximately 44% at 700°C and 900°C. Almost all the hydrogen volatilized, and the nitro-

Moisture content, weight %, wet basis	42
Major components of dry solids, weight %	
Carbon	23.2
Hydrogen	3.80
Oxygen*	50.4
Nitrogen	0.25
Aluminum	6.87
Calcium	6.86
Sulfur	0.03
Silicon	7.05
Titanium	1.53
Heating value, MJ/kg	8.38

*By difference

I. Major elements in the sludge from the secondary fiber plant using duplicate analyses

Element	Amount in dry sludge, ppm	Detection limit, ppm
Cr	40	10
Cu	60	20
Fe	2050	100
Mg	2750	100
Mn	30	10
P	900	200
Sr	50	10
V	30	10
Zn	155	20

II. Minor components in sludge from the secondary fiber plant using duplicate analyses

Ashing temperature, °C	500	700	900
Residue, wt. % of dry sludge solids	51.5	44.2	43.8
Elements in residue, wt. % of element in dry sludge solids			
C	8.4	1.5	0.2
H	1.9	2.0	0.5
Al	99	96	101
Ca	101	100	104
Si	99	96	101
Ti	98	95	100

III. Retention of major components of the sludge in ash produced at 500°C, 700°C, and 900°C with 1 h ashing time

gen in the residue was below the detection limit of 0.1%. By comparison, Table III shows that essentially all the major inorganic species were retained in the residue.

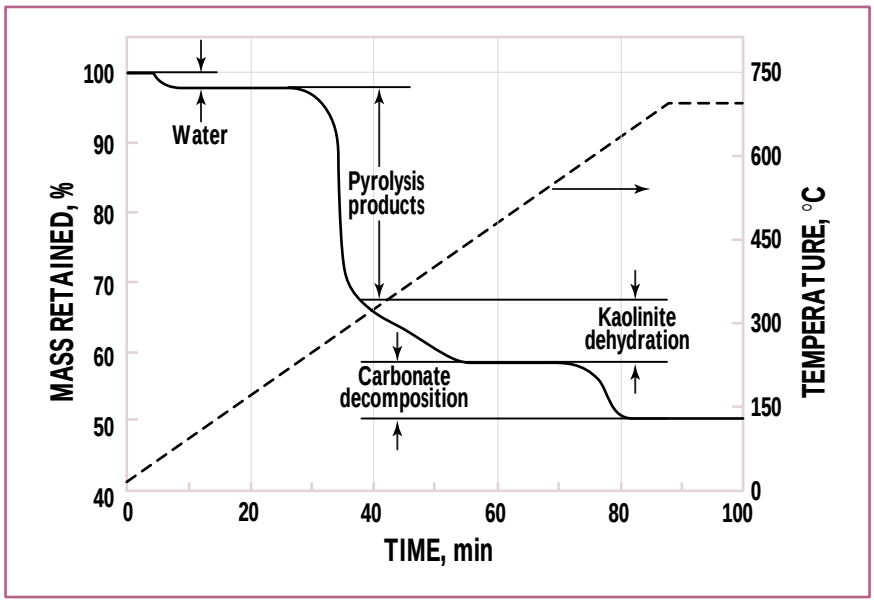
Within the detection limits of the analysis procedure used, essentially all the minor species were retained. The carbon content of the residue produced at 500°C corresponds within experimental error to the carbonate associated with the calcium in the residue as uncalcined $CaCO_3$. The much lower carbon content of the ash at 700°C and 900°C indicates that calcination of the $CaCO_3$ present in the sludge was nearly complete at these temperatures. By comparison, the theoretical ash yields—assuming that only inorganic oxides and carbonates were present—would be 48.9% if the calcium were $CaCO_3$ and 41.3% if it were CaO.

THERMOGRAVIMETRIC MEASUREMENTS

After pulverizing and classifying small samples of the dried sludge, we collected and used the 125–250 µm size fraction for our experiments. We selected this size fraction because it was the largest we could feed into the laminar entrained-flow reactor. Since pyrolysis occurs very rapidly under the experimental conditions employed, we wanted to use the largest possible particles to extend the reaction time over which pyrolysis occurred.

Samples of this material were pyrolyzed in a commercially available thermogravimetric analyzer to obtain pyrolysis rate and char yield data. The pyrolysis experiments were performed in a nitrogen atmosphere at heating rates of 10°C/s, 20°C/s, and 30°C/s. The sample weights were 7–12 mg. Initial and final temperatures were 30°C and 900°C, respectively. A data acquisition computer collected mass vs. time data. Figure 1 shows a typical mass vs. time curve for the thermobalance runs using 9.61 mg dried sludge.

Four mass loss regions are evident in Fig. 1. The first occurs below 100°C and represents the loss of approximately 1.5% moisture from the sample. The second occurring between 225–300°C is a result of the loss of volatile organic (pyrolysis) products from decomposition of the organic matter. The third at 300–440°C is the result of dehydration of kaolinite, $2SiO_2 \cdot Al_2O_3 \cdot 2H_2O$. This material changes its structure to meta-kaolinite, $2SiO_2 \cdot Al_2O_3$ as discussed by Douglas et al. *(3)* and Latva-Somppi *et al. (7)*. The fourth region, at approximately 500–630°C, is probably the decomposition of $CaCO_3$ and $MgCO_3$. These tempera-



1. Mass retained and reaction zone temperature vs. time for a thermogravimetric analysis run at 10°C/s

Region	Measured mass loss, %	Expected mass loss, %
Drying	1.5	-
Pyrolysis	31.2	-
Kaolinite dehydration*	9.7	4.5
$CaCO_3$, $MgCO_3$ decomposition	7.9	8.0

*Based on two waters of hydration per kaolinite

IV. Measured vs. expected mass losses during the thermobalance run in Fig. 1

ture ranges agree with the results presented by Latva-Somppi *et al. (7)*, although our temperature ranges are slightly lower. This may result from the smaller sample size of 7–12 mg used in our experiments vs. the 15mg samples used by Latvasomppi *et al. (7)*.

Table IV shows that the expected mass losses for carbonate decomposition based on the Ca and Mg content of the sludge agree well with the measured mass losses. The expected mass losses for kaolinite dehydration based on the Al and Si content of the sludge is approximately half the measured mass loss between 300–440°C. This may be due to an overlap between the loss of volatile organic matter and kaolinite dehydration. Table V gives the mass loss during pyrolysis for each heating rate.

Using the mass vs. time data from the thermogravimetric analysis runs, a rate equation was determined for devolatilization. We first differentiated the data numerically and then fit it to a rate equation of the form

$$(1/m)(dm/dt) = A \cdot \exp[E_a/RT] \cdot (m/m_o)^b \quad (1)$$

where

m = mass of volatile material remaining in the residue, mg
T = temperature of the sample, °K
R = ideal gas constant
A, E_a, and b = empirical constants.

The values from fitting the data from the runs at all three heating rates are $A = 7933\ min^{-1}$, $E_a/R = 6544$ K, and $b = 0$. The predicted and experimental rates agreed well as shown in Fig. 2. The mean square difference between the measured and calculated rates, defined as $\Sigma[(r_{exp} - r_{calc})^2/n]$, was 0.0019. In calculating the mean square difference, n is the total number of data points taken. It includes all three heating rates and all reaction times.

GAS AND CHAR PRODUCTS FROM PYROLYSIS AT HIGH HEATING RATES

Samples of the 125–250 µm dried, pulverized sludge solids underwent pyrolysis in nitrogen at high heating rates (above 10^3 °C/s) in a laminar entrained flow reactor (LEFR). The design and operation of the LEFR is described elsewhere *(8)*. We fed the dried sludge particles to the reactor at a rate of 0.6–0.8 g/min. The particles were suspended in the unheated primary gas flow stream. The secondary flow stream entering the reactor concentric to the primary flow was preheated to the reactor temperature before entering the reactor. Table VI shows the operating conditions for these experiments. There were two separate runs at each condition.

Solid and gaseous products exiting the heated zone of the reactor underwent quenching with nitrogen in a water-cooled sample probe. We separated the solids by size in a cyclone with a 3 µm particle diameter cutoff with collection of finer particles on a 0.8 µm cutoff filter. We

collected, weighed, and analyzed the cyclone catches (chars) for carbon and metals. We analyzed the filter catches for Al, Si, P, S, Ca, and Ti using Electron Dispersive x-ray analysis (EDAX). The product gases for carbon-containing gas species using a Fourier Transform Infrared Spectrometer (FTIR), and NO_x species were analyzed by a chemoluminiscence meter

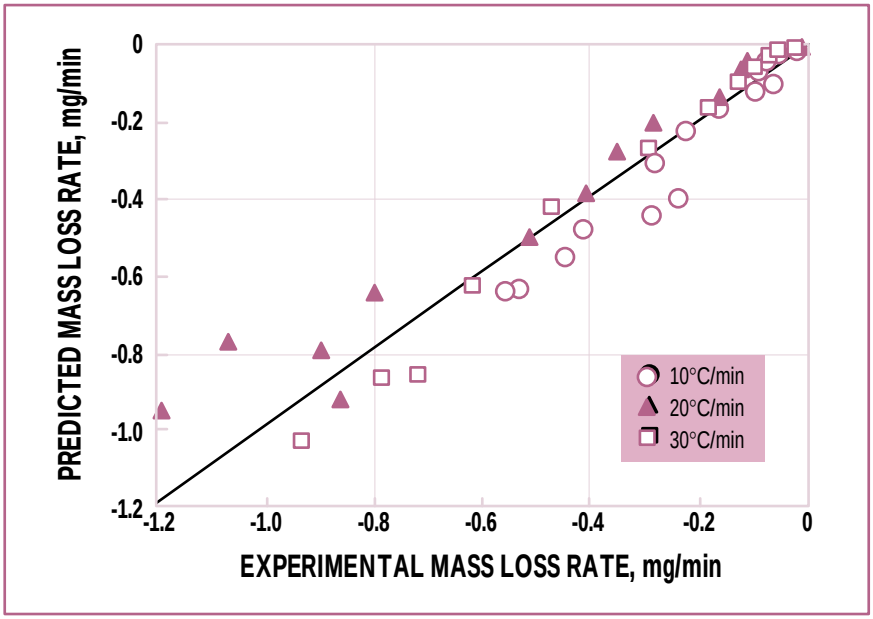
Table VII shows that for the LEFR runs at 500°C and 700°C, the char yields were higher than those from the data in Table V. This indicates that pyrolysis and calcination of $CaCO_3$ may not have been complete in the short residence times available. Much carbon volatilized as acetaldehyde at all three temperatures and as hydrocarbon gases at 900°C. The fraction of carbon measured as CO and CO_2 increased as reaction temperature increased.

Table VIII shows the range of NO_x values measured at the three different temperatures. The conversion of fuel N to NO_x increased rapidly with increasing temperature. The fraction of the fuel nitrogen converted to NO_x was always low, however.

There was little solid residue collected on the post-cyclone filter—typically about 1% of the material fed to the LEFR. Table IX shows that EDAX scans for Al, Ca, Si, Ti, S, and Cl indicated that the main elements were Al, Si, and, Ca. The ratios of Ca, Si, and, Ti to Al in these samples were nearly the same as in the dried sludge (see Tables I and II.). Only the Si/Al ratio was different—approximately 30% higher in the filter catch samples.

GASIFICATION OF THE CHAR RESIDUE

We conducted limited experiments in which dry sludge solids were first pyrolyzed in nitrogen in a fixed bed reactor, and the char carbon was then gasified with water vapor. In these experiments conducted at 500°C, 700°C, and 900°C, essentially all the fixed carbon in the residue from pyrolysis achieved gasification within one hour. There were no kinetic data collected in these experiments.



2. Measured vs. experimental rates of pyrolysis for dry sludge

Heating rate, °C/min	Pyrolysis mass loss, % of dry sludge
10	31.7
20	30.6
30	30.3

V. Mass loss during pyrolysis vs. heating rate for thermobalance experiments

Reactor temperature, °C	500	700	900
Primary flow rate, m^3/s	$8.3x10^{-7}$	$8.3x10^{-7}$	$8.3x10^{-7}$
Secondary flow rate, m^3/s	$3.3x10^{-4}$	$3.3x10^{-4}$	$3.3x10^{-4}$
Particle residence time, s	1.4	1.1	0.9

VI. Operating conditions and results from the laminar entrained-flow reactor experiments with 125–250 µm dried sludge particles

ASH EVALUATION

An additional sludge sample weighing approximately 50 kg and containing 53% by weight dry solids underwent slow gasification with air in a semi-batch pilot furnace at the Albany Research Center, Bureau of Mines, laboratory in Albany, OR. The ash produced had a bulk density of 378 kg/m^3.

Differential Scanning Calorimetry (DSC) and thermogravimetric (TGA) analyses of the ash from the pilot semi-batch gasification run (see Fig. 3) showed endotherms between 380–440°C and between

Run	1	2	3	4	5	6
Temperature, °C	500	500	700	700	900	900
Solid residues, % of feed mass						
In cyclone	83.7	77.9	74.2	70.0	56.2	58.8
On post-cyclone filter	1.2	1.4	0.5	0.6	0.8	0.8
Carbon in gases, % of carbon in dry sludge solids						
CO	0.3	0.5	1.1	1.3	35.4	33.2
CO_2	0.6	0.8	0.3	0.2	5.7	5.0
CH_4	0.2	0.1	0.2	0.2	3.6	3.6
C_2H_4	0.0	0.0	0.1	0.1	5.9	5.9
CH_3OH	0.0	0.0	0.0	0.0	0.2	0.1
C_2H_2	0.0	0.0	0.0	0.0	1.3	1.2
CH_2O	0.3	0.4	0.6	0.7	3.2	3.0
C_2H_4O	5.0	8.7	10.7	12.6	13.9	13.0
Total C in gases	6.4	10.6	13.1	15.1	69.0	64.9
Carbon in cyclone catch, % of catch		22.0	18.1		10.5	
Carbon in cyclone catch, % of C input		76.6	56.4		26.1	
Carbon recovered, % of C in feed*		85.1	70.4		93.1	

*These experiments did not collect the tars produced

VII. Solid residue and gas yields and analyses from the laminar entrained-flow reactor experiments with 125–250 µm dried sludge particles.

Run	Temperature, °C	NO_x, % of fuel N
1	500	0.23
2	500	0.21
3	700	0.50
4	700	0.74
5	900	-
6	900	8.2

VIII. NO_x yields from pyrolysis of sludge in the LEFR runs

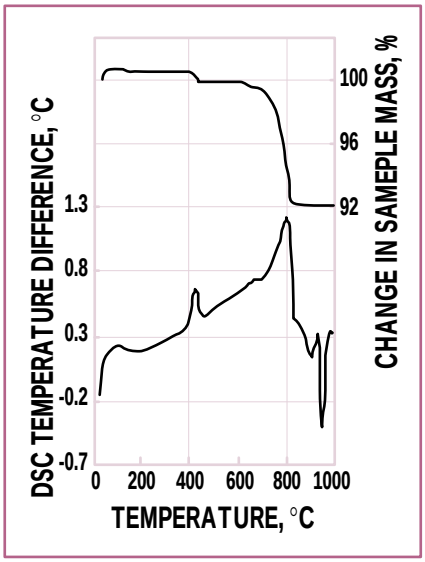
Element	500°C	700°C	900°C
Al	28	27	28
Ca	24	19	25
Si	37	33	37
Ti	4.8	4.7	4.8
S	1.7	6.0	1.5
Cl	4.7	11	2.8

IX. Relative amounts of several elements in the filter catch samples determined by EDAX analysis

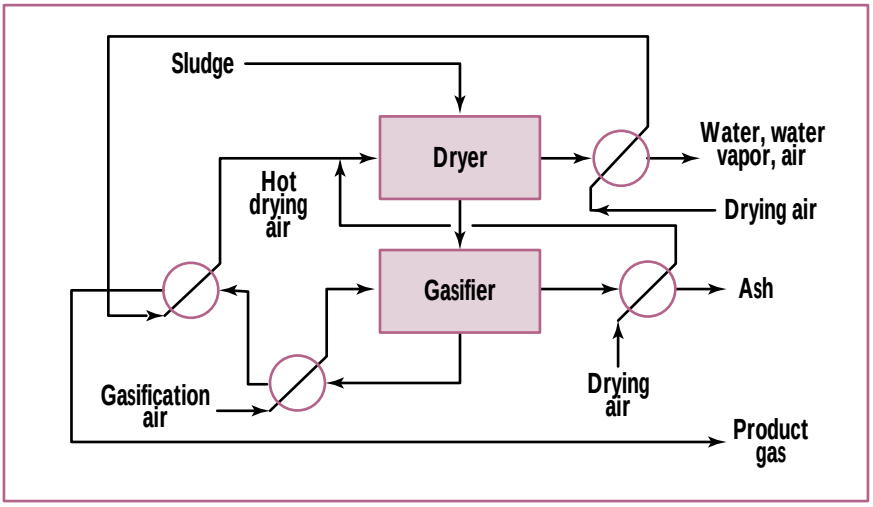
700–810°C with transition to a glassy phase above 810°C. The endotherms correspond to sample mass losses on the TGA curve. They are dehydration of clay and calcination of $CaCO_3$ and $MgCO_3$, respectively, as discussed earlier. The phase transition at approximately 940°C corresponds to the endothermic transformation of meta-kaolinite into an alumina-silica spinel *(7)*. The endotherm beginning at 860°C and peaking at 900°C may be the melting peak for Na_2SO_4.

We collected approximately 10 kg of the ash and sent it to the Oregon Department of Transportation for evaluation as a mineral admixture for Portland cement concrete. Table X includes the results from the evaluation and analyses. The table also includes the Al_2O_3, SiO_2, Fe_2O_3, and SO_3 content of the ash determined in a separate analysis.

The standards shown in Table X are for Class C fly ash produced from lignite or subbituminous coal *(9)*. No standards currently exist for ash from combustion or gasification of sludges from secondary fiber plants. When evaluated using the lignite or subbituminous coal standards, the ash tested met the specifications for strength activity index, soundness,



3. Differential scanning calorimetry analysis of the ash from the pilot semi-batch gasification run



4. Conceptual process for gasification of sludge with air

SO_3 content, and moisture content. It failed to meet three specifications: the sum of the oxides Al_2O_3, SiO_2, and Fe_2O_3; fineness; and loss on ignition.

A similar sludge from a mill producing printing paper has had successful use as a fuel for a cement kiln. That sludge also contained less than 50% Al_2O_3, SiO_2, and Fe_2O_3 based on its ash content. The sum of Al_2O_3, SiO_2, and Fe_2O_3 was low by less than 5.7% of 50%. Adding one or more of these oxides to the ash after gasification would meet this specification.

The ash was too coarse to meet the fineness specification. It was unclear from the test results whether this was because the particles were too coarse or whether fine particles agglomerated to increase the apparent particle size. In either case, grinding the ash could remedy this problem.

Failure to meet the specification for loss on ignition was the result of incomplete gasification in the semi-batch pilot furnace. Douglas *et al. (3)* reported losses on ignition of 0.06% and 1.38% in ash from pilot combustion trials with different deinking sludges. Our material was only slightly over the specified value of 6%. Meeting the loss on ignition should not be a problem in a commercial gasifier.

PROCESS CONCEPT

The data obtained in this study indicates it is possible to convert the sludge to a fuel gas for burning elsewhere in the mill and to an inorganic component for concrete or other construction materials. Figure 4 shows a process to convert the sludge. It consists of a sludge dryer, a gasifier, and heat exchangers. The gasifier is a partial air oxidation gasifier. Recovery of energy from the gas products provides preheat for the air entering the reactor and drying for the sludge.

We calculated mass and energy balances for the process in Fig. 4. The calculations are based on the sludge properties listed in Tables I and II. They assumed complete gasification of the carbon and that the gaseous products are at equilibrium with the water gas shift reaction. This assumption is valid at temperatures over 700°C and for gases in the presence of alkali metal catalysts at lower temperatures *(10)*.

The energy balances were calculated based on a reactor operating temperature of 700°C. Using equilibrium calculations performed on the HYSIM Process Simulator *(11)*, the air factor was determined from the stoichiometric oxygen required to completely convert the fuel carbon to gases. Our calculations indicate the available energy from the reaction is greater than that required to maintain the reactor at 700°C. The product streams (fuel gas and ash) were cooled to 110°C. We assumed the heat recovered from these streams after the air preheater and from the reactor was available for drying the wet sludge and for generation of steam. We neglected heat losses from the process. Table XI contains the mass flows for key process streams. Table XII shows the net energy recovered from the process.

Using this basis, an air factor of 0.579 was necessary to operate the reactor at 700°C. The product gas contained 5.4% CO and 17.1% H_2 by volume (dry basis). Its heating value was 2.64 MJ/Nm^3.

The energy required for drying the sludge was calcuated based on a drying air to dry sludge solids mass ratio of 4.0 and energy recovery

Test	Result	Standard*	Test procedure
Chemical composition, wt. %			
Al_2O_3	19.6	50% min	
SiO_2	24.0	(sum of Al_2O_3,	
Fe_2O_3	0.73	SiO_2, and Fe_2O_3)	
SO_3	0.22	5% max	
Loss on ignition	6.36%	6% max	ASTM C114
Moisture content	0.36%	3% max	ASTM C311
Physical characteristics			
Density, g/cm^3	2.86		ASTM C118
Specific gravity	2.98-3.07		ASTM D854
Fineness (325 sieve)	45.7 retained	34% max	ASTM C430
Drying shrinkage	0.013%		ASTM C157
Soundness (autoclave exp.)	0.008%	0.8% max	ASTM C151
Air-entrainment	0.336		ASTM C185
strength			ASTM C109
3-day, psi	2730		
7-day, psi	3820		
28-day, psi	4240		
Strength activity index			
7-day, %	92.27	75% min	
28-day, %	83.30	75% min	
Gilmore time of set			ASTM C266
Initial, min	40		
Final, min	65		
Normal consistency, %	41		ASTM C187

*based on ASTM C618 - 89a *(9)*

X. Evaluation of ash from gasification of dried sludge solids as a mineral admixture for Portland cement concrete.

from the exiting air stream to cool it to 75°C. The energy required to operate the sludge dryer was 204.7 MJ/ton of sludge solids. A net 137.2 MJ/ton dry sludge was available for steam generation.

The thermal efficiency of the process, defined as the heating value of the product gas plus the energy available for steam generation divided by the total energy input (25°C reference temperature), was 69.8%.

We made similar calculations to determine the improvement in heating value of the product gas and process efficiency if elemental oxygen was substituted for some air of the in the gasifier. Table XIII shows the effects on heating value of the product gas, useful energy recovered, and total energy efficiency. The heating value increased by 17% with approximately 30% of the oxygen supplied as elemental oxygen and by 40% with about 60% of the oxygen supplied as elemental oxygen. The total thermal efficiency for this process was about the same regardless of the extent of elemental oxygen substitution. The thermal efficiency of a combustor burning the product gas would be higher for a fuel with a higher heating values because of a lower sensible heat loss with the combustion products stream.

CONCLUSIONS

The sludge investigated in this study had a heating value of 8.38 MJ/kg dry solids. This low value and the difficulty in dewatering fibrous materials to less than 42% moisture content (wet basis) made it a low quality fuel. Its adiabatic flame temperature of 1550°C indicates that it will sustain combustion or gasification by partial oxidation. Experimental measurements indicate that it is easily pyrolyzed and that conversion of carbon to combustible gases by partial combustion would proceed readily.

An evaluation of the ash produced by gasification with air at 700°C showed that the ash did not

Stream	1	2	3	4	5	6	7	8	9
Temperature, °C	25	25	110	650	700	337	100	100	75
Flow rate, kg									
Total mass	166.7	146.0	100.0	146.0	197.6	197.6	197.6	48.4	66.7
C	21.0		21.0						
H	3.7		3.7						
O or O_2	26.9	33.6	26.9	33.6					93.2
N or N_2		112.4		112.4	112.4	112.4	112.4		306.8
Ash	48.4		48.4					48.4	
H_2O (l)	66.7								
CO					10.6	10.6	10.6		
CO_2					60.5	60.5	60.5		
H_2					2.4	2.4	2.4		
H_2O (v)					11.7	11.7	11.7		466.7

l = liquid
v = vapor

XI. Mass and energy flow rates, temperature, and composition for key process streams in Fig. 3 using 100 kg dry sludge solids as the basis

meet ASTM standards for use as a mineral admixture for Portland cement concrete. The ash contained too much carbon, too high a fines fraction, and too little Al_2O_3, SiO_2, and Fe_2O_3 for use as-is for this purpose. Selection of more optimal process conditions for gasification would resolve the first two of these problems. Sludge with a similar shortage of Al_2O_3, SiO_2, and Fe_2O_3 has had commercial use as a fuel for a cement kiln.

Material and energy balances calculated for a process for converting recycled fiber sludge to a low to medium heating value fuel gas and a useful ash indicate that gasification with air could produce a fuel gas with a heating value of 2.64 MJ/Nm^3 (dry basis) (air factor = 0.579) at 700°C. Calculations indicate that the proposed system is feasible and would generate excess energy during operation. The estimate of the total thermal efficiency of the process is 69.8%. Gasification with oxygen-enriched air increases the heating value of the product gas and the thermal efficiency of the combustor burning it. TJ

Heating value of dry product gas	449.0
Energy from reactor	251.0
Energy from cooling product gas	58.7
Energy from cooling ash	32.1
Energy to dryer	204.7
Net thermal energy available, MJ	137.1
Heating value of dry sludge solids	839.6
Efficiency, %	69.8

XII. Energy balance for the process in Fig. 4 with the gasifier operating at 700°C showing energy values as MJ/100 kg dry sludge solids

All authors are with Oregon State University, Department of Chemical Engineering, Gleeson 103, Corvallis, OR 97330 except Lundy, who is with Department of Civil Engineering at the same university and O'Connor, who is with Albany Research Center, Bureau of Mines, U.S. Department of Interior, Albany, OR.

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Fraction of O_2 supplied as elemental oxygen	0	0.295	0.584
Air ratio	0.579	0.583	0.586
Heating value of dry product gas, MJ/Nm^3	2.64	3.08	3.70
Total energy recovered, MJ/100 kg sludge solids	586.1	587.6	588.7
Efficiency, %	69.8	70.0	70.1

XIII. The effect of substituting elemental oxygen for air on product gas heating value and overall process thermal efficiency

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