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Co-firing High Sulfur Coal with Refuse Derived Fuels
Technical Progress Report #5

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1. Summary

Studies involving the tubular furnace are in the process of identifying the ideal experimental coal-to-refuse derived fuel (RDF) ratio for use in the AFBC system. A series of experiments with this furnace has been performed to determine the possible chemical pathway for formation of chlorinated organic compounds during combustion of various RDF sources. Phenol and chlorine appear to be likely reactants necessary for the formation of these compounds. The main goal of these experiments is to determine the exact experimental conditions for the formation of chlorinated organic compounds, as well as methods to inhibit their development.

Work on the fluidized bed combustor has involved five combustion runs, in which a combustion efficiency of greater than 96% and with a consistent CO₂ concentration of approximately 13% was obtained. Modifications responsible for these improvements include the addition of the underbed fuel feed system and revision of the flue gas sampling system. New methods of determining combustion efficiency and percentage of SO₂ capture using TG techniques to analyze combustion products are being developed. The current outlook using this TGA/FTIR method is very promising, since previously obscured reactions are being studied. The analysis of combustion products is revealing a more complete picture of the combustion process within the AFBC system.

2. Studies with the Tubular Furnace

The main focus of the continued studies with the tubular furnace is to identify the organic compounds evolved during combustion of coal with components of municipal solid waste (MSW). TG/FTIR and TG/MS studies have indicated some interaction between the components, at both the slow and fast heating rates. The results show that at a fast heating rate, the decomposition reactions are compressed into a narrow time range. Hence, the decomposition stages cannot be separated, as with the slow heating rate. The FTIR data indicate that more hydrocarbons are produced at a fast heating rate, whereas more oxidation products are obtained at the slow heating rate. Phenol was identified from the combustion of newspaper and RDF (refuse derived fuel). Mass spectrometric results indicate that molecular chlorine (Cl_2) is produced concurrently with HCl during the decomposition of PVC. The ratio of HCl/ Cl_2 is around 150. This suggests that some of the abundant HCl molecules may be undergoing the Thermal Deacon Reaction. This reaction is favored up to 600°C at atmospheric pressures. The Deacon reaction could be a plausible starting point for the formation of chlorinated dioxins from the combustion of chlorine rich fuel mixtures as is postulated below:¹⁻³

It is difficult to identify chlorophenol using the TG/MS system for two reasons. First, the amount of phenol ($m/z = 94$) in the gas phase is very small. Secondly, the chlorophenol has the same parent mass, 128, as naphthalene, and at very low levels the isotope $\text{ClC}_6\text{H}_4\text{OH}$ ($m/z = 130$). This problem can be resolved using the tubular furnace/gas trap/GC/MS system.

Prior to conducting any studies, the furnace was calibrated for accurate temperatures in order to reproduce reaction conditions exactly. A list of parameters which could vary (and hence effect the results) was also identified.

The raw materials, including coal 92073 (high sulfur, low chlorine), PVC, newspaper, cellulose and RDF were burned in the furnace, and the evolved gases were collected in a trap of methylene chloride cooled in an ice bath. A heating tape (400°C) was wrapped around the transfer tube, where it was not in contact with the furnace, to minimize condensation.

Four grams of coal 92073 was burned in 1 standard cubic foot per hour (SCFH) of air. The furnace was preheated to a temperature of 850°C before the sample was introduced. The resulting gases were trapped in the chilled CH_2Cl_2 . Compounds tentatively identified by GC/MS analysis are listed in Table 2. [*Tentative* identification in this report means simply a match of the mass spectrum of a compound with that of a compound in the NIST mass spectra library.]

Similar experiments were conducted using PVC, newspaper, cellulose and RDF. The results of the analysis are listed in Table 2. It could be seen from the results of replicate analyses that phenol is one of the components evolved during the combustion of coal, newspaper, cellulose, and RDF. One of the major components of PVC combustion is HCl. This is in agreement with the TG/MS results and the proposed mechanism for the formation of polychlorinated phenols. It now remains to investigate the exact ratio of coal to RDF and the experimental conditions under which they form. A sample of synthetic RDF was studied using the same conditions as that for the raw materials. The results of this study are given in Table 3.

Studies were done on mixtures of PVC and RDF. The following three parameters were altered and their effects were investigated: (a) Ratio of PVC to RDF, (b) Temperature, and (c) Air flow (amount of oxygen). The results of this study are given in Table 4. They do not indicate any specific trend, making it difficult to draw conclusions and inferences for further study.

In parallel with the above work, a synthetic approach was used. The object here was the identification of chlorinated species formed from phenol and HCl and the conditions under which they survive. Phenol (100 mg) was burned in the presence of 0.5% Cl_2 in N_2 and the evolved gases trapped in methylene chloride and analyzed using GC/MS. The results are summarized in Table 5. The temperature of the reaction was varied, yet the results seemed fairly consistent. It was possible to identify phenol, chlorophenol and dichlorophenol, though the absence of heating tape caused partial condensation of monochloroisomers. The same reaction performed in the solution phase seemed to occur readily at room temperature (Table 6A). It then remained to resolve the issue of a vapor phase or solvent phase reaction. If a reducing agent for molecular chlorine (Cl_2) is used, then the chlorine is immediately reduced on reaching the solvent, forestalling reaction in the solution phase. The reducing agent used was NaHSO_3 . In order to facilitate solubility of the salt, it was prepared in an acidic aqueous solution, serving a two-fold purpose: (1) reducing molecular chlorine when it comes in contact with solution and (2) reducing the solubility of chlorinated phenols (if any are formed in the vapor phase).

The resulting aqueous solution (in the gas trap) was then extracted using 10 mL aliquots of methylene chloride to partition aqueous and organic phases. This greatly reduced

the possibility of reaction in aqueous phase, so it would be reasonable to conclude that the compounds identified in subsequent analyses were formed due to reaction of vapors. The results obtained are shown in Table 6B.

The focus of further studies using the tubular furnace will be to establish exact experimental conditions for the formation of these chlorinated compounds and for the possible condensation of these compounds to form dioxins (PCDDs). The exact composition of materials forming the dioxins will also be determined. It then remains to find conditions, means and techniques for reducing or preventing the formation of PCDDs and PCDFs in the AFBC (Atmospheric Fluidized Bed Combustor) system.

3. Fluidized Bed Combustion

Since the last quarterly report, five combustion runs have been performed in the AFBC system. The dates of these experiments were August 15, October 17, October 19, November 10, and November 30. The overall goal of these experiments has been to attain 96% combustion efficiency with a CO₂ concentration of 15%. Western Kentucky University coal sample #95011 (high sulfur/low chlorine) and Kentucky limestone have been used in all experiments thus far. Experiments that are underway using the tube furnace apparatus, as discussed in the previous section, will determine the ideal experimental conditions (combination of coal and other fuels) for the burning of refuse derived fuels (RDF) with the AFBC system.

Improvements in combustion efficiency have been made with the addition of the underbed feed system, as illustrated in Figure 1. This modification, by which fuel and bed

material is injected three inches above the setter plate, enables the mixture to be thoroughly blended with the existing combusting material. Hence, better combustion efficiency and more flexibility with air flow rates can be attained. Combustion efficiencies greater than 96% are being routinely obtained since this modification was made.

In working towards the desired 15% CO_2 concentration, a variety of problems have arisen. The foremost of these has been leakage in the flue gas sampling line, which creates a decrease in the concentration of CO_2 due to increased atmospheric air intake. Finding these leaks has been difficult, since the lines only leaked significantly when placed in certain positions. For example, significant leaking might take place when changing the ion chromatograph (IC) sampling bottles. This problem has been corrected by building a platform next to the combustor and placing the GC instrument approximately 3 feet away. This has reduced the number of possible leaking connectors from eleven down to two and has reduced the length of sampling line from 80 feet down to 3 feet. During the last combustion run of November 30, a consistent CO_2 concentration of approximately 13% was attained. Although this is slightly below our desired goal, it represents the first time that consistent (Figure 2) and reliable data (FTIR spectra confirmed) were attained. Efforts during that combustion run to saturate the only possible leaking connectors with pure CO_2 showed no increase in CO_2 and no decrease in N_2 . These results provide strong evidence that no leaks are occurring. Current plans for increasing the concentration of CO_2 involve: changing limestone/air/coal feed rates, reducing the bed height during combustion while maintaining constant temperature, and eliminating purge air sources (used to maintain a clear viewing window inside the combustor).

During this reporting period the GC and FTIR equipment has also been automated so that the flue gas is analyzed precisely at preset times. This has resulted in a reduction of the number of people actually gathering and handling data, and hence, a reduction in errors.

Methods to utilize thermal analysis equipment in determining combustion efficiency are also currently being developed. This idea came from a paper presented at in 1989 at the 18th Annual NATAS Conference by S.A. Mikhail and A.M. Turcotte⁴ entitled "A TG Method of Analysis for Use in Monitoring the Efficiency of Fluidized Bed Coal Combustion Processes." This idea was modified to take advantage of our on-line TG/FTIR instruments in order to obtain a more complete picture of the combustion processes. The TG methods are capable of not only monitoring combustion efficiency, but also of determining the amount of sulfates that are absorbed by the bed material. This particular aspect is most interesting since it provides an additional way to measure the goal of 90% capture of SO_2 . Results from the TG technique for measuring combustion efficiency will be compared with conventional methods prior to implementation. Further investigation is underway. Fly ash from each combustion run will also be investigated using the TG/FTIR method.

The experimental conditions for analysis of bed material consist of the following six steps.

1. Heat at $20^\circ\text{C}/\text{minute}$ to 850°C in N_2 .
2. Hold the 850°C temperature for 10 minutes in N_2 .
3. Hold the 850°C temperature for 10 minutes in 40% O_2 with the balance N_2 .
4. Hold the 850°C temperature for 5 minutes in 5% H_2 with the balance N_2 .
5. Heat $10^\circ\text{C}/\text{minute}$ to 1000°C in 5% H_2 with the balance N_2 .

6. Hold the 1000°C temperature for 20 minutes in 5% H₂ with the balance N₂.

As an example, using the TG curve in Figure 3, there is a weight loss from the bed material sample that occurs at 400°C due to dehydroxylation of Ca(OH)₂. Figure 4, which is an FTIR spectrum, supports this reaction by showing the formation of H₂O during this time frame. The following reaction is suggested:

More information about the reactions of the bed material can be gained by again examining the TG curve in Figure 3. Between 500°C and 800°C another significant weight loss occurs, which is mainly due to the thermal decomposition of CaCO₃. This reaction is supported by the data in Figure 4, which shows the production of CO₂. During step 3 another weight loss is noted, due to combustion of remaining carbon material. This evolution is likewise supported by the emission of CO₂ (second peak) and H₂O during this step, as shown in Figure 4. Steps 5 and 6 involve the reduction of CaSO₄ to CaO, with the suggested reaction:

The curves given in Figure 4 are consistent with this reaction.

The FTIR results show the evolution of SO₂ in two stages. The first one is between 600°C and 750°C, and the second one is above 800°C. This second peak is due to the reduction of CaSO₄. The first smaller peak might be due to the decomposition of CaSO₃.

Thus, the second weight loss at stage 2 is not only due to the decomposition of CaCO₃, but also to the decomposition of CaSO₃. Therefore, the TG/FTIR results are needed to calculate

the amount of SO_2 evolved in order to determine the amount of CaSO_3 present in this stage.

Further investigation is underway.

In conclusion, much time and effort is being expended to attain the goals of 96% combustion efficiency with a 15% CO_2 concentration. This principally involves:

1. Developing and utilizing techniques (TG/FTIR/MS and conventional carbon methods) which accurately measure the true combustion efficiency of the AFBC system.
2. Obtain consistent and reliable data from the flue gas sampling systems and on-line analysis systems.

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5. Tables

Table 1. Capillary GC Parameters Set for GC/MS Analysis

Injector Temp. (°C): 250

Oven Conditions:

Initial Temp. (°C): 100

Initial Time (min): 5

Temp. Program:	Rate (°C/min)	Temp. (°C)	Time (min)
	5	150	5
	5	200	5
	5	250	5
	6	310	5

Equil. Time (min): 1

Flow Controller Parameters:

Carr. Gas Press. (kPa): 106.50

Carr. Gas Press Time (min): 5

Press. Program:	Rate (kPa/min)	Press (kPa)	Time (min)
	1.30	122.30	5
	1.20	135.90	5
	1.30	147.50	4
	1.20	159.10	5

Total Flow (mL/min): 71

Split Ratio: 32

Interface Temp. (°C): 290

**Table 2. Tentative Identification by GC/MS of Compounds Produced
During the Combustion of Raw Coal**

Run 1	Coal
Sample:	4 g of coal
Airflow:	1.0 SCFH of air
Isothermal:	850°C (preheated to 850°C)
Heating tape:	45 volts
Date:	8/10/95
Coolant:	ice
Solvent:	225 mL of methylene chloride
Results:	trimethylbenzene, phenol , methyl nonane, undecane, propenylbenzene, methyl-phenol, ethylxylene, cyclopentane, dimethylphenol, tetrahydronaphthalene, dodecane, naphthalene, methylethylphenol, tridecane, tetradecene, methylnaphthalene, dimethylnaphthalene, hexadecane, heptadecane, hexadecene, eicosane
Run 2	PVC
Sample:	4 g of PVC
Airflow:	1.0 SCFH of air
Isothermal:	850°C (preheated to 850°C)
Heating tape:	45 volts
Date:	8/11/95
Coolant:	ice
Solvent:	225 mL of methylene chloride
Results:	toluene, xylene, ethylbenzene, chlorobenzene, styrene, methyl-ethylbenzene, propylbenzene, trimethylbenzene, indene, methylindane, naphthalene, methylnaphthalene

Table 2. (continued)

Run 3   Newspaper

Sample: 4 g of newspaper
 Airflow: 1.0 SCFH of air
 Isothermal: 850°C (preheated to 850°C)
 Heating tape: 45 volts
 Date: 8/16/95
 Coolant: ice
 Solvent: 225 mL of methylene chloride
 Results: toluene, furfural, furfuryl alcohol, butenol, furanone, styrene, methylcyclopentanone, 5-methyl-furfural, **phenol**, benzofuran, methylphenol, indene, dimethylphenol, methoxy-methylphenol

Run 4   Cellulose

Sample: 4 g of cellulose
 Airflow: 1.0 SCFH of air
 Isothermal: 850°C (preheated to 850°C)
 Heating tape: 45 volts
 Date: 8/18/95
 Coolant: ice
 Solvent: 225 mL of methylene chloride
 Results: furfural, **phenol**, cyclohexanone, methylfuran, furanone, methylphenol, 5-methylfurfural, methylphenol, furfuryl alcohol, methylundecane, dianhydroglucopyranose

**Table 3. Tentative GC/MS Identification of Compounds Evolved
During the Combustion of RDF**

Sample: 4 g of RDF
Airflow: 1.0 SCFH of air
Isothermal: 850°C (preheated to 850°C)
Heating tape: 45 volts
Date: 8/22/95
Coolant: ice
Solvent: 225 mL of methylene chloride
Results: toluene, acetic acid, cyclopentanone, cyclooctene, hexanal, octanal, furfural, furfuryl alcohol, furanone, styrene, cyclohexanone, 5-methylfurfural, **phenol**, decene, decane, methylphenol, indene, dimethylphenol, methoxy-methylphenol, dodecane, ethyl-methoxyphenol, methyl-propenylphenol, hexadecene, pentadecene, pyrene, benz[a]anthracene, hexadecenol

Table 4. Tentative GC/MS Identifications from Blends

Run 6

Sample: 2.5 g of coal + 2.5 g of PVC
Airflow: 1.0 SCFH of air
Isothermal: 850°C (preheated to 850°C)
Heating tape: 45 volts
Date: 8/29/95
Coolant: ice
Solvent: 225 mL of methylene chloride
Results: toluene, octene, octane, chlorobenzene, methylundecene, ethylbenzene, xylene, styrene, nonane, trimethylbenzene, propenylbenzene, propylbenzene, **phenol**, decene, methylphenol, indene, butylbenzene, methylindan, tetrahydronaphthalene, dimethylphenol, naphthalene, methylnaphthalene, biphenyl, dimethylnaphthalene, acenaphthylene, methylbiphenyl, fluorene, methylanthracene, phenylnaphthalene, pyrene, methylpyrene, benz[a]anthracene

Run 7

Sample: 2.5 g of PVC + 2.5 g of RDF
Airflow: 1.0 SCFH of air
Isothermal: 850°C (preheated to 850°C)
Heating tape: 45 volts
Date: 9/04/95
Coolant: ice
Solvent: 225 mL of methylene chloride
Results: toluene, furfural, acetic anhydride, **chlorobenzene**, ethylbenzene, xylene, furanone, styrene, cyclohexanone, 5-methylfurfural, benzaldehyde, propylbenzene, **phenol**, α -methylstyrene, trimethylbenzene, indene, 3-methylphenol, furfuryl alcohol, decene, naphthalene, biphenyl, acenaphthylene, fluorene, anthracene, methylanthracene, octadecene, pyrene, benz[a]anthracene

Table 4. (continued)

Run 8	Varying amount of PVC and RDF
Date:	9/20/95
Sample:	2.5 g of PVC + 10.0 g of RDF
Airflow:	1.0 SCFH of air
Isothermal:	850°C (preheated to 850°C)
Heating tape:	45 volts
Coolant:	ice
Solvent:	225 mL of methylene chloride
Results:	No phenols, no chlorobenzene [but see runs 7, 9-12]
Run 9	Varying air flow
Date:	9/27/95
Sample:	2.5 g of PVC + 10.0 g of RDF
Airflow:	2.0 SCFH of air
Isothermal:	850°C (preheated to 850°C)
Heating tape:	45 volts
Coolant:	ice
Solvent:	225 mL of methylene chloride
Results:	phenol , methylphenol
Run 10	Varying air flow
Date:	10/09/95
Sample:	2.5g of PVC + 10.0g of RDF
Airflow:	3.0 SCFH of air
Isothermal:	850°C (preheated to 850°C)
Heating tape:	45 volts
Coolant:	ice
Solvent:	225mL of methylene chloride
Results:	toluene, furfural, ethylbenzene, xylene, styrene, cumene, 5-methylfurfural, phenol , α -methylstyrene, decene, trimethylbenzene, propylbenzene, indene, methylphenol, furanmethanol, dodecane, benzoic acid, naphthalene

Table 4. (continued)

Run 11   Varying amount of (PVC+RDF)

Date: 10/13/95

Sample: 2.5 g of PVC + 5.0 g of RDF

Airflow: 2.0 SCFH of air

Isothermal: 850°C (preheated to 850°C)

Heating tape: 45 volts

Coolant: ice

Solvent: 225 mL of methylene chloride

Results: benzene, cyclohexene, toluene, heptene, furfural, ethylbenzene, styrene, xylene, 5-methylfurfural, **phenol**, propylbenzene, indene, furfuryl alcohol, naphthalene, methylnaphthalene, biphenyl

Run 12   Varying combustion temperature

Date: 10/18/95

Sample: 2.5 g of PVC + 2.5 g of RDF

Airflow: 2.0 SCFH of air

Isothermal: 450°C (preheated to 450°C)

Heating tape: 45 volts

Coolant: ice

Solvent: 225 mL of methylene chloride

Results: benzene, toluene, furfural, xylene, ethylbenzene, styrene, 5-methylfurfural, **phenol**, methylethylbenzene, indene, furfuryl alcohol, methylheptene, naphthalene, methylnaphthalene, ethylnaphthalene

Table 5. Study of Reactions Between Chlorine and Phenols

Run 13

Sample: 100 mg of phenol
Date: 10/25/95
Gasflow: >65mL/min of 0.5% Cl₂ in N₂
Isothermal: Room to 400°C
Heating tape: N.A.
Coolant: N.A.
Solvent: 225 mL of methylene chloride
Results: phenol, *p*-chlorophenol and *o*-chlorophenol

Run 14 repeat Run 13

Sample: 100 mg of phenol
Date: 10/27/95
Gasflow: >65mL/min of 0.5% Cl₂ in N₂
Isothermal: Room to 400°C
Heating tape: N.A.
Coolant: N.A.
Solvent: 225 mL of methylene chloride
Results: phenol, *p*-chlorophenol, *o*-chlorophenol and 2,4-dichlorophenol

Run 15 Varying temperature

Sample: 100 mg of phenol
Date: 10/27/95
Gasflow: >65mL/min of 0.5% Cl₂ in N₂
Isothermal: Room to 250°C
Heating tape: N.A.
Coolant: N.A.
Solvent: 225 mL of methylene chloride
Results: phenol, *p*-chlorophenol, *o*-chlorophenol and 2,4-dichlorophenol

Table 5. (continued)

Run 16   Varying heating rate

Sample: 100 mg of phenol
Date: 10/31/95
Gasflow: >65mL/min of 0.5% Cl₂ in N₂
Isothermal: 250°C (preheated to 250°C)
Heating tape: N.A.
Coolant: N.A.
Solvent: 225 mL of methylene chloride
Results: 2,4-dichlorophenol

Run 17   Adding heating tape and set temperature constant

Sample: 100 mg of phenol
Date: 10/31/95
Gasflow: >65mL/min of 0.5% Cl₂ in N₂
Isothermal: 250°C (preheated to 250°C)
Heating tape: 30 volts (400°C)
Coolant: N.A.
Solvent: 225 mL of methylene chloride
Results: phenol, 2,4-dichlorophenol

Table 6. Chlorination in the Vapor and Solvent Phases

(A) Not using a reducing agent

Run 18 Determining if reaction take place in trap liquid

Sample: 100 mg of phenol dissolved in methylene chloride

Airflow: >65mL/min of 0.5% Cl₂ in N₂ goes through the solvent

Isothermal: Room Temperature

Heating tape: N.A.

Date: 11/04/95

Coolant: N.A.

Solvent: 225 mL of methylene chloride

Results: phenol, *p*-chlorophenol, *o*-chlorophenol and 2,4-dichlorophenol

Run 19 Repeat of run 18

Sample: 100 mg of phenol

Airflow: >65 mL/min of 0.5% Cl₂ in N₂

Isothermal: 250°C (preheated to 250°C)

Heating tape: 30 volts (400°C)

Date: 11/11/95

Coolant: N.A.

Solvent: 225 mL of methylene chloride

Results: phenol, *p*-chlorophenol, *o*-chlorophenol, and 2,4-dichlorophenol

Table 6. (continued)

(B) Using a reducing agent (NaHSO_3)

Run 20 Trap liquid contains reagent to destroy Cl_2 , preventing reactions in trap

Sample: 100 mg of phenol

Airflow: >65 mL/min of 0.5% Cl_2 in N_2

Isothermal: 250°C (preheated to 250°C)

Heating tape: 30 volts (400°C)

Date: 11/14/95

Coolant: N.A.

Solvent: 2 g of NaHSO_3 dissolved in 50 mL of acidified water

Extraction: Five times with 10 mL of methylene chloride each time.

Results: phenol, o-chlorophenol, 2,4-dichlorophenol, a trichlorophenol

Run 21 Repeat of run 20

Sample: 100 mg of phenol

Airflow: >65 mL/min of 0.5 % Cl_2 in N_2

Isothermal: 250°C (preheated to 250°C)

Heating tape: 30 volts (400°C)

Date: 11/19/95

Coolant: N.A.

Solvent: 2 g of NaHSO_3 dissolved in 50 mL of acidified water

Extraction: Five times with 10 mL of methylene chloride each time.

Results: phenol, o-chlorophenol, 2,4-dichlorophenol

6. Figures

Figure 1. Drawing of the AFBC combustor.

Figure 2. Gas sampling data (CO₂) concentration vs. time).

Sample: BEDING.102

Size: 10.1825 mg

Method: FIRST BLOOD

Comment: RAMP20C/MIN, T0750C N2; ISO 20MIN, N2/02; 20C/MIN TO 1000 N2/H2

TGA

File: A: BEDING.001

Operator: SHEN

Run Date: 16-Nov-95 08:29

Figure 3. TG curve for bed material.

Absorbance / Wavenumber (cm⁻¹)

File # 1 = FBC004#74 @ 3957.431

bedding 10-19-95, N2 200ml/min to 850C iso 10, N2/O2 10, H2/N2 5, 10c/min to 1000C iso 20min 500mg

See 11

11/26/95

Figure 4. FTIR spectrum for bed material.

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