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Co-firing high-sulfur coals with refuse-derived fuel 1

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Abstract

The fundamental thermal behavior of five materials (Illinois coal #6, Kentucky coal #9, polyvinyl chloride, cellulose and newspaper) has been investigated using the TGA/FTIR/MS system under different combustion conditions. At a fast heating rate, the decomposition temperatures shift towards higher temperatures and the maximum weight loss rates increase by 3-7 times those at the slow heating rate. The gases evolved from the decomposition have been analyzed kinetically. More organic compounds are identified at the faster heating rate. Also, molecular chlorine is observed in the oxidation of PVC. These two species may lead to the formation of chlorinated organic compounds. The results indicate that the TGA/FTIR/MS system can be used to evaluate the effect on the environment of co-firing high-sulfur coal with refuse-derived fuels.

Keywords: Co-firing; Evolved gas analysis; TGA/FTIR/MS

1. Introduction

The Environmental Protection Agency reported that the total municipal solid waste (MSW) produced in the US increased from 179 million tons in 1988 to 195 million tons in 1990 [1]. It is predicted that the country will produce about 216 million tons of garbage in the year 2000 [2]. The amount of waste generated and the rapidly declining availability of sanitary landfills have forced most municipalities to evaluate alternative

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waste management technologies for reducing the volume of waste sent to landfills. The fraction of MSW that was processed by technologies such as separation and recycling, composting, and waste-to-energy is forecast to increase from a few percent today to 30 40% by the year 2000 [3].

Waste-to-energy conversion of MSW appears to be most attractive because of the energy recovered, the economic value of the recycled materials, and the cost saving derived from reduced landfill usage. However, extra care needs to be taken in burning MSW or Refuse-Derived Fuel (RDF) to optimize the operating conditions of a combustor so that the combustion takes place in an environmentally acceptable manner. For instance, polychlorinated dibenzodioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) have been found in the precipitator fly ash and flue gas of a number of incinerator facilities in the United States and Europe. Though the amount of PCDDs and PCDFs is only in the parts per billion to parts per trillion range, these chlorinated organics exhibit very high toxicity $(LD_{50} < 10 \,\mu g - Kg^{-1})$ and 2,3,7,8-tetrachlorodibenzo-dioxin has been found to be acnegenic, carcinogenic, and teratogenic. This has slowed or even stopped the construction and operation of waste-to-energy plants.

Some important studies have been conducted to reduce the release of chlorinated organics. Recently, Lindbaurer [4] reported that co-firing an MSW combustor with 60% coal drastically reduced the PCDD/PCDF levels. Sulfur in coal was found to be a major factor in the drastic reduction of PCDD/PCDF formation, even at S/CI ratios as low as 0.64 [5]. Additional research is needed to develop optimum co-firing conditions and parameters. Investigations are also needed to determine why and how the co-firing of high-sulfur coals with MSW can reduce the emissions of polychlorinated organic compounds.

In this study, we report the thermal behavior of two coals, polyvinyl chloride (PVC), cellulose and newspaper. The PVC, cellulose and newspaper can be used to formulate a synthetic refuse-derived fuel (RDF).

2. Experimental

Two coal samples, (sample numbers 90003 and 92073) were used in this study. Analyses are shown in Table 1. The samples of each coal were prepared by pulverizing a one-kilogram split of each coal to -60 mesh (-250 microns) and further splitting with a Brinkman Model PTZ 8-position sample divider. Splits of the -60 mesh materials were used for characterization and analysis, as well as for thermal analysis studies.

The noncombustibles of MSW such as metals, ceramics and glass will be excluded from this study. Thus, newspaper (shredded sample from the WKU Coal and Fuel Laboratory), medium molecular weight polyvinyl chloride (PVC) homopolymer resin (Oxy225, Occidental Chemical Corporation) and cellulose (Whatman Corporation) were selected for formulating a simulated synthetic RDF (the combustible portion of MSW).

The TG/FTIR system consists of a DuPont 951 Thermogravimetric Analyzer interfaced with a Perkin-Elmer 1650 Fourier Transform Infrared Spectrometer. In our TG/FTIR experiments, samples of ~ 100 mg (except PVC where 30 mg was used) were

| Parameter | 90003 (IL#6) | 92073 (KY#9) | PVC | Newspaper | Cellulose |
|-------------------------|--------------|--------------|--------------------|-----------|-----------|
| % Moisture ^a | 8.76 | 9.88 | 0.00 | 4.21 | 3.12 |
| $%$ Ash | 8.12 | 26.34 | 0.36 | 4.12 | 0.00 |
| % Vol. matter | 34.25 | 32.56 | 100 | 81.21 | 96.88 |
| $%$ Fixed-C | 57.63 | 41.10 | 0.00 | 0.00 | 0.00 |
| $%$ Carbon | 75.07 | 59.53 | 38.71 | 45.67 | 43.44 |
| % Hydrogen | 5.21 | 3.50 | 4.20 | 6.67 | 6.80 |
| % Nitrogen | 1.64 | 1.17 | 0.07 | 0.00 | |
| % Sulfur | 1.26 | 4.44 | 0.22 | 0.05 | 0.02 |
| $\%$ Oxygen | 8.32 | 4.99 | 0.00 | 43.12 | 49.76 |
| Chlorine, ppm | 3800 | 289 | 56.45 ^b | 260 | 0.00 |
| Heating value | 13.428 | 10.334 | 8556 | 7741 | 6723 |
| (Btu/lb) | | | | | |
| Rank of Coals | B | C | | | |

Table 1 Proximate and ultimate analysis data for raw materials

^a Moisture is as-determined. All other analysis are reported on a dry basis. The rank for each coal is high volatile A, B, or C bituminous coal. ^b The unit of chlorine in PVC is percent.

heated in air (50 ml min⁻¹) at a slow heating rate of 10°C min⁻¹ to 700°C, and at a fast heating rate of 100° C min⁻¹ to 900° C, then held for 5 min at the maximum temperature. Using the TG/FTIR analytical system, the spectra and profiles of gaseous species evolving from the TG system were recorded and analyzed using Grams 386 software. The TG/MS system consists of a DuPont Instruments Model 951 Thermogravimetric Analyzer interfaced with a VG Thermolab Mass Spectrometer. In our TG/MS system, the mass detection range is 1-300 atomic mass unit (amu). The electron impact ionization energy is 70 e. A sample size of \sim 20 mg was used in all runs. The same heating rates and air flow rate as in the TG/FTIR system were adopted. The MS system can automatically sample the gases evolved from the TG system, analyze them by its quadrupole analyzer, and save the mass spectra. The detailed experimental conditions are discussed in Lu's thesis [6].

3. Results and discussion

3.1. TG/DTG results

Comparisons of the TG curves at the slow heating rate for the five raw materials, coal 92073, coal 90003, PVC, newspaper, and cellulose, are shown in Fig. 1. The TG curves reveal some distinguishing characteristics of the five raw materials:

(i) The coals have much higher decomposition temperatures and show weight loss rates much slower than PVC, newspaper and cellulose.

(ii) Coal 92073 has a lower decomposition temperature than coal 90003 despite their similar volatile matter contents. The difference is about 30°C. This may be due to

Fig. 1. TG curves for the raw materials at slow heating rate.

the lower rank of coal 92073. The devolatilization stage and the oxidation of fixed carbon in the DTG curves overlap one another for both coals.

(iii) Coal 92073 has a higher residue than 90003, This is consistent with the proximate analysis data (Table 1).

(iv) PVC shows three weight-loss phases. The initial weight loss (64%) between 250 and 400°C is clearly mainly due to the loss of HC1. This can be inferred from the percentage (58%) of HC1 in PVC and is confirmed by FTIR data. The second weight loss between 400 and 500°C, and the third weight loss between 500 and 600°C, are due to main-chain fragmentation and to the oxidation of the carbon residue.

(v) The newspaper curve has three phases. The first one between 25 and 120°C is due to moisture that is attached to the polymer chain. The second weight loss between 220 and 400°C, and the third weight loss between 400 and 500°C, are due to decomposition of the polymer structure in newspaper. It is not decomposed completely, possibly owing to the presence of mineral fillers.

(vi) Like newspaper, cellulose also shows three phases: (i) loss of moisture; (ii) oxidation of volatiles; (iii) combustion of carbon residues.

Fig. 2 is a comparison of TG curves at the fast heating rate for the five individual materials. The curves are similar to those obtained at the slow heating rate (Fig. 1). However, two important changes take place at the faster heating rate. One is that the weight loss curves shift from lower temperatures to higher ones. The other is that the rates of weight loss increase.

The data show that the maximum rates of major weight loss (R_{max}) increase 3-7 times and the decomposition temperatures (T_{max}) at major R_{max} increase by about 40-80°C at COMPARISON OF TG CURVES FOR RAM MATERIALS AT FAST HEATING RATE
Files:H90003.024 H92073.023 HPVC.026 HDEWSPAPER. FINEWSPAPER. 014 HCELLULOSE.013

Fig. 2. TG curves for the raw materials at fast heating rate.

the fast heating rate. Plots of temperature vs. time also indicate that the heating rate remains constant during the runs. This indicates that the decomposition reactions of fuels occur much faster and at higher temperature as the heating rate increases. The decomposition at the slow heating rate was studied in order to understand the mechanism of each stage; the study at the fast heating rate was to approach the conditions of the Atmospheric Fluidized Bed Combustor (AFBC) unit. The heating rates in an AFBC system are much faster than those in TGA experiments. Thus, we can expect that decomposition reactions would occur at higher T_{max} and greater R_{max} in AFBC systems.

3.2. FTIR results

Fig. 3 shows the three-dimensional FTIR spectra of PVC decomposition products at the slow heating rate. In the figure, the vertical axis is in absorbance units. The horizontal axis is in wavenumbers $(cm⁻¹)$. The perspective axis is in time (s). In considering the heating rate, the time scale can correspond to the decomposition temperature. For the slow heating rate, the spectra were collected beginning at 100° C of TGA and scanning at a speed of one per minute. So the first spectrum curve obtained corresponds to the temperature of 100° C in the TG curves. For the fast heating rate, the first spectrum corresponds to the initial (room) temperature of the TGA curve.

At the slow heating rate, a notable phenomenon is observed at 674 cm^{-1} in Fig. 3. The profile at this wavenumber has two maxima. One is at about $300^{\circ}C (Z = 1200)$; the other is at about 500° C (Z = 2400). There are two possible compounds absorbing at

Fig. 3. Three-dimensional FTIR spectrum of PVC at slow heating rate.

this wavenumber: carbon dioxide and benzene [7]. The absorbance at this wavenumbet is often attributed to carbon dioxide. At the first maximum, however, there is no absorbance from CO₂ at 2356 and 3600-3700 cm⁻¹. This indicates that this absorbance is not from CO_2 . Combined with the peaks at 1500 and 3050 cm⁻¹, we can conclude that the first maximum could be due to absorbance by benzene. This is also supported by MS results. HCl (2798 cm^{-1}) and benzene are first released at 230–380°C $(Z = 800-1600)$ and reach their maximum at approximately 300°C ($Z = 1200$), with some organic acids (1700-1800 cm⁻¹) and alcohols (1000-1200 cm⁻¹, maximum at $Z = 1200$) from partial oxidation of the low molecular weight PVC. This is in accordance with the TG/DTG results for the first weight loss. When the temperature is increased, the evolution (maximum at $Z = 2500$ or 530° C) of CO, carbon dioxide and water was observed, with the emission (2800–3050 cm⁻¹, maximum at about $Z = 2000$ or 430°C) of methane and some hydrocarbons (2800–3050 cm⁻¹). This indicates that oxidation is predominant at higher temperatures. The emission of methane and hydrocarbons corresponds to the second weight loss in TG/DTG. However, the combustion of the PVC skeleton chains occurs over a large range of temperatures from 300 to 600°C, which is shown by the maxima ($Z = 2500$ or 530°C) of the profiles of carbon dioxide and carbon monoxide, respectively. This corresponds to the third weight loss in its TG-DTG curve. These results imply that the PVC is not readily flammable at lower temperatures, owing to its chlorine content.

Fig. 4 shows the 3D-FTIR spectra of PVC combustion at the fast heating rate. The gaseous products are evolved in a much narrower range of time $(Z = 200-800$,

Fig. 4. Three-dimensional FTIR spectrum of PVC at fast heating rate.

300-900 $^{\circ}$ C, compared to Z = 500-3000, 190-600 $^{\circ}$ C for the slow heating rate). Thus, the three distinct decomposition stages observed at the slow heating rate were not observed at the fast heating rate. This results in the synchronous evolution of hydrocarbons, HC1, and organic and inorganic oxidation products. Fig. 5 shows the 3D-FTIR spectra of newspaper at the slow heating rate. The FTIR spectra of cellulose are very similar to those of newspaper. A number of peaks between 250 and 500°C are observed that correspond to the release of water, $CO₂$ and CO. These are large peaks owing to the larger oxygen content and hydroxy functionalities in the molecules of these materials. Also, compared to PVC, many more organic acids (mainly formic and acetic acids) are produced during the combustion of newspaper and cellulose. These can be identified in the three-dimensional spectra by groups of peaks at 2500-3400 (OH), 1700-1800 (C=O), 1033 cm⁻¹ for methanol, 1106 cm⁻¹ for formic acid and 1175 $cm⁻¹$ for acetic acid. The appearance of these products can be attributed to the poly-hydroxyl structures in newspaper and cellulose. The additional characteristic product identified in the spectra of both newspaper and cellulose is formaldehyde. This compound is difficult to identify because its two characteristic absorbance bands, $C=O$ and $C-H$, are overlaid with those of other products. In the range 1700- 1800 cm^{-1} , the resolution is not enough to separate the peaks from different carbonyl groups. Therefore the identification is possible only by its C-H stretch vibration at around $2700-2900 \text{ cm}^{-1}$. This requires use of a fine structural spectrum to distinguish it from the stretching vibrations of C-H and H-CI bonds. As the heating rate increases

Fig. 5. Three-dimensional FT1R spectrum of newspaper at slow heating rate.

字 500
0

1500 1000

3000

2500 2000

4000 3500 3000 2500 2600 1500 1000 500

Ahsorbance / Wavenumber (cm-1)

5

1

0 ~'

from slow to fast, the spectra of newspaper and cellulose display a compressed change similar to that of PVC.

The spectra of coal 92073 (Fig. 6) at the slow heating rate shows a similar trend. However, there are still considerable differences between the spectra of the two coals. There are more water peaks in 90003 than in 92073 in the regions from 1300 to 1700 $cm⁻¹$ and 3500 to 4000 cm⁻¹. This can be attributed to more volatile matter, and oxygen and hydrogen content in 90003. The COS (2073 cm⁻¹) and SO₂ (1374 cm⁻¹) peaks are stronger for coal 92073 than 90003, reflecting the higher sulfur content. The profiles of SO₂ peaks in 92073 provide some evidence about the sulfur forms present. The aliphatic sulfur decomposes first at $280-350^{\circ}$ C; then pyrite and thiophene rings decompose at 350-500°C; and finally sulfate salts are predominant at $500-700$ °C. The peaks at 1777 and 1167 cm⁻¹ can be attributed to acetic acid by comparing their shape and wave number with standard spectra.

Fig. 7 shows 3D-FTIR spectra of coal 92073 at the fast heating rate. Unlike the three peaks that appear in the profile of sulfur dioxide at slow heating rate, only one overlapping peak of sulfur dioxide is observed, along with other gases, at the fast heating rate. This change indicates that the oxidative decomposition of different sulfur forms occurs in a narrow temperature range except for sulfate sulfur, which decomposes continuously and slowly over a long range. The 3D-FTIR spectra of coal 90003 at the fast heating rate are shown in Fig. 8. In comparison with the slow heating rate, an important change is observed in that water, CO, methane and other organic products

Absorbance / Wavenumber (cm-1)

Fig. 6. Three-dimensional FTIR spectrum of coal 92073 at slow heating rate.

Fig. 7. Three-dimensional FTIR spectrum of coal 92073 at fast heating rate.

Fig. 8. Three-dimensional FTIR spectrum of coal 90003 at fast heating rate.

are evolved very early. This may be supported by the release of more hydrocarbon gases and water (dehydrogenation or incomplete combustion of fuels) at the initial decomposition stage. It is notable that at the fast heating rate, the hydrocarbons are released at almost the same time as HC1, while at the slow heating rate, they are evolved at higher temperatures than HCI.

Table 2 is a summary (not including water peaks which appear in every sample) of the tentative identification of FTIR peaks for the five raw materials at the slow and fast heating rates. More than 15 gaseous species can be identified. The data indicate that at the fast heating rate it is easier to find methane and xylenes in the evolved gases. At the slow rate, more oxidation products such as acids and alcohols are found.

From all the FTIR data, it is demonstrated that more hydrocarbons are produced at the fast heating rate whereas more oxidation products are obtained at the slow heating rate. This may be due to incomplete, combustion of fuels at the fast rate. More importantly, it is observed that the chlorine and hydrocarbon species formed during the combustion are released simultaneously at the fast heating rate. Hence, these results indicate there are more opportunities and greater possibility for yielding harmful chlorinated organic compounds during co-firing coals with refuse-derived fuels at the faster heating rate that occurs in an AFBC system. However, this conclusion should be supported by the results from the combustion of blends of coal and RDF which will be pursued in a further study.

Table 2
Tentative identification of FTIR peaks for raw materials (wavenumber/cm⁻¹)^a Ientative identification of FTIR peaks for raw materials (wavenumber/cm⁻¹⁾^{a} $-284/1006$ $+161$

171

The "H-" prefix indicates the fast heating rate. "News" and "Cell" represent newspaper and cellulose, respectively.

3.3. MS results

Because many species in the gaseous mixture from combustion cannot be identified or even detected by FTIR, the MS was used to complement the FTIR. As with the FTIR, MS results from the fast heating rate show a similar trend, in that the decomposition reactions are compressed into a narrow time range during the heating process. So the decomposition stages of different components cannot be separated. This makes our analysis complicated although it simulates the combustion conditions in the AFBC system more closely. Therefore, we attempt only to compare and discuss the changes resulting from different heating rates, and finally try tentatively to identify some of the major mass peaks for each MS result.

Fig. 9 shows the profiles of some of the peaks in the MS spectrum of coal 92073. The profiles of peaks m/z 18, 32, 44, 60, and 64 can be attributed to the ions of H₂O, O₂, $CO₂$, COS, and SO₂, respectively. The water profile shows three peaks. The first peak at around 100°C is from the free moisture in coal. The second and third peaks accompanied by other gaseous products are produced due to the combustion and decomposition of coal. The oxygen profile remains stable up to about 350°C, after which it decreases due to its consumption in the combustion of the coal. The small inverse peak indicates a maximum consumption of oxygen. In addition, sulfur dioxide shows three peaks. This result is the same as that obtained in the FTIR spectra. The carbonyl sulfide has a mass of 60 amu which is the same as that of acetic acid. Thus the MS results cannot distinguish between acetic acid and carbonyl sulfide. However these two species can be distinguished from FTIR results. The FTIR spectrum of 92073 has shown that acetic acid was released from 200 to 400°C (maximum at about 300°C which corresponds to the first peak in the MS profile), and carbonyl sulfide was evolved from 250 to 570°C (maximum at about 370°C, which corresponds to the second peak in the MS profile).

Fig. 9. MS peak profile of coal 92073 at slow heating rate.

Fig. 10. MS peak profile of PVC at slow heating rate.

Fig. 10 is the profile of some peaks for PVC at the slow heating rate. These peaks are identified as HC1 (mass 36), molecule chlorine (mass 70) and chlorobenzene (mass 112), and their isotopic peaks (38 for H³⁷Cl, 72 for ³⁵Cl–³⁷Cl, 74 for ³⁷Cl₂ and 114 for C₆H₅– 37° Cl). They appear at around 300° C, spanning the same temperature range. Also, the ratios of their isotopic peak areas are close to the theoretical chlorine isotope fraction. The same results can be obtained from PVC at the fast heating rate. All of this mass spectrometric evidence shows that chlorine molecules are produced concurrently with the strong HCl release. The ratio of $HC1/C1₂$ is around 150. The existence of chlorine molecules suggests that some fraction of the abundant HC1 could be undergoing a thermal Deacon reaction. At atmospheric pressures, this reaction is thermodynamically favored up to about 600°C. Occurrence of the Deacon reaction

 $4HCl + O_2 \rightarrow 2Cl_2 + 2H_2O$

can be supported by the pyrolysis results of PVC. Fig. 11 shows the profiles of mass peaks 36, 38, 70, 72, 112 and 114 during the pyrolysis of PVC in helium. The profiles of mass 36 and 38 indicate HC1 production. Chlorine molecules were not obtained at 250-350°C due to lack of oxygen as an oxidation reagent. The Deacon reaction could provide a plausible starting point for the formation of chlorinated dioxins from the combustion of chlorine-rich fuel mixtures. However, additional evidence is needed to confirm this possibility.

Fig. 12 shows us that benzene *(m/z* 78) and naphthalene *(m/z* 128) only appear at about 300°C. This is supported by FTIR and TGA results of PVC as discussed above. All other mass peaks show two or three decomposition stages in their profiles. These are consistent with the three decomposition stages in the TGA results of PVC. At about

Fig. 11. MS peak profile of PVC during pyrolysis.

Fig. 12. MS peak profile of PVC.

300°C, most of the other peaks in the mass spectrum contribute a fraction to the first weight loss in the TG experiments in addition to HC1, benzene, and naphthalene. These peaks also make contributions to the second weight loss noted in the TG experiments at around 430°C. For the third weight loss around 540°C in the TG experiments, carbon dioxide is the major product along with a small amount of water. The maximum in the water profile occurs around 450°C. This indicates that most hydrogen in PVC

Table 3
Tentative parent structures of MS peaks for raw materials^a lentative parent structures of MS peaks for raw materials^a

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has been released before 540° C in the forms of HCl, H₂O and organic compounds. These conclusions can also be supported by FTIR results. From the profiles *ofm/z* 182, 146, and 92, we can see another maximum at about 370°C. These cannot be identified from TG/FTIR results. This indicates that mass spectrometry is more sensitive than TG/FTIR techniques.

Cellulose is a polysaccharide. Therefore it is expected that the major products include a series of furan derivatives, which are common products from the thermal decomposition of saccharides, including 3-methyl-2-furanone and/or furfuryl alcohol (98), furfural (96), methylfuran (82) and furan (68). Table 3 summarizes the tentative identification of major peaks for the mass spectra of five individual components at both heating rates. There are more than 20 detectable organic compounds in evolved gases. Most of them are aromatic compounds. The compounds in bold type are also confirmed by GC/MS analysis of the evolved gases after trapping in methylene chloride.

4. Conclusions

The TG/FTIR/MS system was used to identify molecular chlorine, furan, phenol and other aromatic compounds, along with HCl, CO, CO_2 , and H_2O in the gaseous products of the combustion of coal, PVC resin, newspaper, and cellulose in air. This is a significant finding that will lead us to examine this combustion step further to look for the formation of chlorinated organic compounds.

The combination of TG/FTIR and TG/MS offers complementary techniques for the detection and identification of combustion products.

The TG/MS techniques allow the study of reaction pathways for the formation of gaseous products during combustion.

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