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An Experimental Investigation into Fast Pyrolysis of Biomass Using an Entrained-Flow Reactor

Mark Bohn
Charles Benham



SERI

Solar Energy Research Institute

A Division of Midwest Research Institute

1617 Cole Boulevard
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AN EXPERIMENTAL INVESTIGATION
INTO FAST PYROLYSIS OF BIOMASS
USING AN ENTRAINED-FLOW REACTOR

MARK BOHN
CHARLES BENHAM

FEBRUARY 1981

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PREFACE

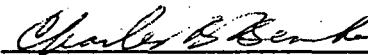
This report describes laboratory experiments which are a part of research in the Solar Thermal Research Branch on the SERI Biomass-to-Diesel process. The series of experiments described here was completed in early 1980 and was undertaken primarily to determine pyrolysis gas composition resulting from fast pyrolysis of agricultural waste feedstocks. Of primary concern was the influence of reactor variables such as temperature and residence time on the gas composition. In the Biomass-to-Diesel process the pyrolysis gases are catalytically converted to liquid hydrocarbon fuels, e.g., diesel. Data presented in this report will support development of full-scale pyrolysis equipment for this process.

The assistance of G. Bessler in design and construction of the apparatus, the assistance of R. Kemna in construction and operation of the apparatus, and the assistance of P. Bergeron in gas chromatographic analysis are appreciated.

Careful review of a draft of this report by Prof. Michael Antal and Prof. James Kuester is gratefully acknowledged.



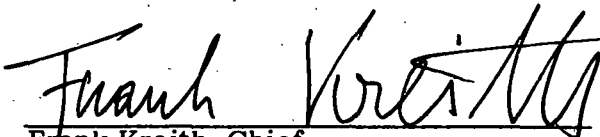
Mark Bohn
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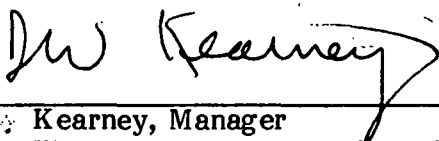
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SECTION 1.0

INTRODUCTION

Destructive distillation (pyrolysis) of biomass materials is an ancient art which has become the object of renewed interest during the past few years. This renewed interest stems from the continuing energy dilemma and the realization by many scientists and engineers that biomass is a viable feedstock for producing fuels and chemicals which are now derived from petroleum feedstocks.

The thrust of the effort within the Solar Thermal Research Branch at SERI is toward producing diesel fuel using gases obtained from an entrained-flow pyrolysis reactor. The feedstocks of primary interest are agricultural nonfood materials such as wheat straw and corn stalks. A catalytic process for converting olefins, carbon monoxide, and hydrogen to liquid hydrocarbons was described by Kuester (1979).

Since the hydrocarbon yield from a catalytic process is influenced by the relative amounts of hydrogen, carbon monoxide, and unsaturated hydrocarbons, it is important for us to understand the interrelationship of the large number of pyrolysis variables which influence gas composition.

Much of the effort in pyrolysis during the past few years has been directed towards exploratory studies of the pyrolysis mechanism.* Welker (1970) presents a review of the literature on pyrolysis and ignition of cellulosic materials. Typically, these studies involved small samples of a given material heated at a few hundred degrees centigrade per minute and they resulted in gas composition, char yield, and kinetics data. An entrained-flow reactor has the advantage over other techniques of inducing continuous pyrolysis with heating rates on the order of 1000°C/s .

Several researchers have reported data from entrained-flow pyrolysis reactors. Finney and Garrett (1973) reported high yields (~ 20 wt %) of ethylene using a finely divided municipal solid waste feedstock in a steam carrier flow, a reactor temperature of about 800°C , and a short residence time (~ 1 s). Brink (1973) studied pyrolysis of wood, kraft black liquor, and municipal solid waste in a reactor having a heating rate of about 1000°C/s , a maximum temperature of 840°C and a residence time of about 3 s. The carrier gas used was nitrogen. Rensfelt (1978) studied pyrolysis of a variety of feedstocks including wood, straw, municipal solid waste, peat, coal, and graphite. The reactor was capable of heating rates to 1000°C/s and gas-phase temperatures were varied from 500°C to 1000°C . Carrier gases used included steam, hydrogen, and nitrogen. Ethylene yields obtained using municipal solid waste were comparable to those obtained by Diebold with pyrolysis tests on municipal solid waste feedstock (ECO-II) which is a commercially available material from Combustion Equipment Associates, New York. In the Diebold (1980) experiments, pyrolysis temperatures ranged from 700°C to 800°C , residence times were from 50 to 200 ms, and ratios of carrier flow to that of biomass varied from 1:1 to 5:1. A combination of steam and carbon dioxide was generally used as the carrier gas. Three runs were made using nitrogen in place of carbon dioxide. The two primary results obtained from this study were that large amounts of olefins (~ 20 wt %) were obtained in the pyrolysis gases and that the greater the dilution

*See Antal (1977 and 1979a), Baker (1975), Broido (1975), Lewellen (1976), Lipska (1966), and Stamm (1956).

of the biomass with carrier gas, the greater the yield of olefins. In a separate study Diebold (1980a) used the same apparatus to obtain data using pure cellulose and birch flour as feedstocks. These materials produced less than one-half the amount of olefins produced using the ECO-II feedstock. McFarland (1972) obtained about 4 wt % ethylene using large particles (1.25 cm) of municipal solid waste in an entrained-flow reactor operated at about 800°C.

In spite of the data obtained from these previously mentioned studies, what the important variables are and what influence these variables have upon gas composition has not been determined. Therefore, the experimental work described in this report was undertaken to provide quantitative information in answer to these questions and to generate the data needed for the design of full-scale equipment.

SECTION 2.0

THE SERI ENTRAINED-FLOW PYROLYSIS REACTOR

As mentioned in the previous section, one of the goals of the SERI program is to make more extensive entrained flow reactor measurements than reported in previous works. Data have been gathered to determine the effects on pyrolysis gas composition of reactor length, reactor temperature, and the ratio of carrier flow to biomass flow. In this section we describe the SERI entrained flow reactor, instrumentation, and experimental procedures.

As shown in Fig. 2-1, biomass is metered into a mixing chamber by a screw feeder where it is entrained by a flow of superheated steam. Water flow rate delivered to the boiler/superheater is measured by a rotameter and the temperature of the steam entering the mixer is also monitored. For smooth delivery, the water is supplied to the boiler/superheater by a piston tank pressurized to about 14 atmospheres (1.4 MPa) by nitrogen.

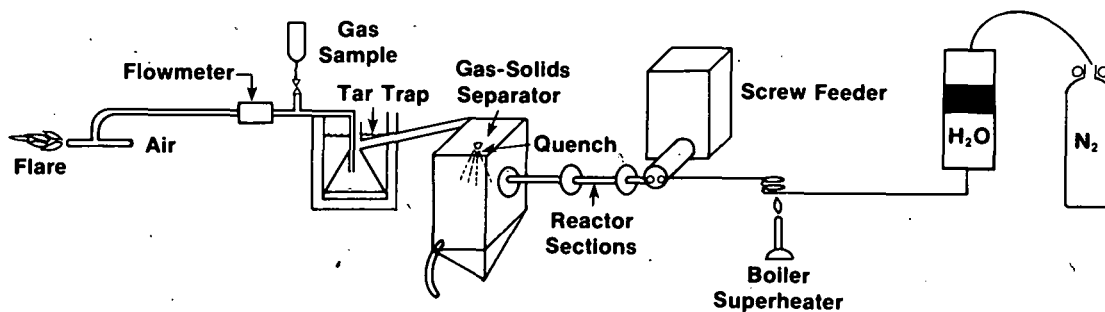


Figure 2-1. ENTRAINED-FLOW PYROLYSIS REACTOR

The entrained biomass/steam flow enters the reactor which is made from electrically heated Vycor glass or stainless-steel tubes with 13 mm inside diameter. Electrical heating has been provided either with a tube furnace for the Vycor tube or by nichrome coils placed near the steel reactor tube. For the Vycor tube, reactor temperature was determined by the temperature setting chosen on the furnace. For the steel tube, power input to the nichrome coils was provided by variacs and the temperature was monitored by chromel/alumel thermocouples. The thermocouples are supported by small steel tubes welded perpendicular to the reactor and held in close contact to the reactor tube by high-temperature ceramic cement. Reactor length is adjusted from 15 to 150 cm by adding or removing reactor sections. Power supplied by the variacs was measured.

Downstream of the last reactor section, a cold water spray quenches the reaction and condenses the steam carrier. At this point, pyrolysis gases and solid particles are separated. The gases then pass through two tar traps consisting of dry ice/water baths, and samples can be collected in small gas bottles downstream of the tar traps. A flow-meter section then measures pyrolysis gas molecular weight and mass flow before the gases are flared with a bunsen burner. The entire system is operated slightly below atmospheric pressure due to suction provided by the air ejector. The gas samples from the bottles are

analyzed with a Hewlett-Packard model 5840 gas chromatograph, which yields data on hydrocarbons through C₅. This chromatograph uses a thermal conductivity detector and helium carrier gas. The three columns (bis-2 methoxy ethyl adipate, porapak Q, mole sieve) are operated at 60°C. Data presented in this report are only for pyrolysis gas composition. At this stage of the research, pyrolysis gas flow rate instrumentation had not been fully debugged and therefore, percent gasification can not be reported.

Flow rate of the biomass particles (500 to 1000 g/h) is measured by a mass balance upon which the screw feeder rests. Temperature is measured by the reactor wall temperature probes and in the gas stream at several stations along the reactor by chromel/alumel thermocouples immersed in the flow through the reactor wall. The probes are made from 0.005 in. wire and are gold plated to minimize emissivity and thereby reduce sensitivity to radiation from the reactor wall. All data are recorded by a microcomputer-based data acquisition system. This system also controls the steel reactor temperature by closing a feedback loop between the wall temperature probes and stepper motors which turn the variacs. Gas sampling ports are also provided at several stations along the reactor length to allow one to measure gas composition as a function of extent of reaction.

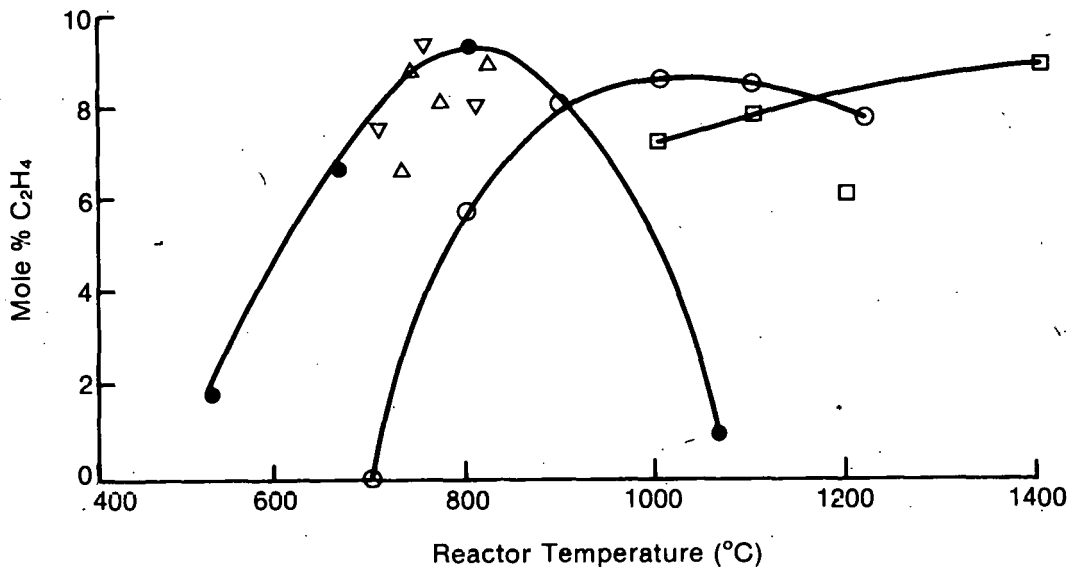
Typically, the reactor and the steam are brought up to temperature together. When steady conditions are reached, the screw feeder is turned on and the reactor temperature is allowed to re-equilibrate to the original temperature. At this time gas samples are taken. A run of about five minutes is required to allow the feed rate of solids to be accurately determined, but for purposes of debugging feed and entrainment problems, runs of thirty minutes have been used.

SECTION 3.0

REACTOR RESULTS

For purposes of comparing results from the SERI reactor with previous experiments (Rensfelt 1978 and Diebold 1980a), we ran experiments with ECO-II fuel as the biomass feedstock. Temperatures from 700°C to 1200°C were used with a 90-cm reactor and temperatures from 1000°C to 1400°C with a 30-cm reactor. Steam-to-biomass ratio was approximately 1:1. The ECO-II particles have an average size of 240 μm.

Diebold (1980a) has shown that steam/biomass ratio can strongly influence ethylene yield. We have taken data for 1:1 ratio from the SERI reactor and from Diebold (1980a). Rensfelt (1978) does not report the steam/biomass ratio used in those experiments. The feedstock used by Rensfelt was not ECO-II but a municipal waste with a composition of $C_6H_{9.3}O_{4.2}$, while ECO-II is reported by Diebold (1980a) to have a composition of $C_6H_{9.8}O_{3.3}$. In addition, particle sizes may have been somewhat larger as the size reported by Rensfelt was " 0.5 mm." For consistency, residence times were calculated for the SERI reactor according to the method used by Diebold (the reactor volume divided by the volumetric flow rate of gases in the reactor at gas exit temperature) while residence times used by Rensfelt were reported to be ~0.4 s.



Reference	Steam Bio	Residence Time
□ Present Study	1 : 1	0.012 s
○ Present Study	1 : 1	0.037 s
△ Diebold (1980 a)	1 : 1	0.05 s
▽ Diebold (1980 a)	1 : 1	0.15 s
● Rensfelt (1978)	?	0.4 s

Figure 3-1. ETHYLENE YIELD VERSUS REACTOR TEMPERATURE AND RESIDENCE TIME

Volume fraction of ethylene is shown in Fig. 3-1. The continuous curves are the data for the SERI 90-cm and 30-cm reactors and from Rensfelt while the individual points are from Diebold (1980a). Absolute scaling of data from the latter is difficult because carbon dioxide was used as a carrier and gas composition was reported on a carbon dioxide-free basis.

Figure 3-1 shows a well defined trend (except for the Diebold data) of ethylene peaks occurring at low temperatures for longer residence times. A decrease in ethylene yield at high temperatures is generally regarded a result of secondary gas-phase reactions which convert the unsaturated hydrocarbons to saturated hydrocarbons of higher molecular weight. The dependence on residence time in an entrained-flow reactor has not been shown before, but can be explained in terms of heat-transfer limitations.

Species evolution from the particles depends on the rate of increase of particle temperature and the heating period (residence time). A short reactor operating at a given temperature could produce a gas composition similar to that produced by a longer reactor operating at a lower temperature. The exception would be the olefins which may be produced in greater volume fraction in the short reactor due to the higher heating rate. This appears to be the case in Fig. 3-1. The ethylene peak for the 30-cm reactor is higher than that for the 90-cm reactor. Note that the longest residence time peak (from Rensfelt 1978) is even higher but a direct comparison of yields from Rensfelt should not be made because steam-to-biomass ratio is unknown for these data. More data for other residence times and other steam-to-biomass ratios should confirm this dependence of gas composition on residence time and temperature and could lead to a correlation between these three parameters.

This same trend is exhibited in Figs. 3-2, 3-3 and 3-4, in which gas composition is plotted as a function of reactor temperature for the 90-cm reactor, for the 30-cm reactor, and for the data from Rensfelt (1978), respectively. The trends, especially for carbon monoxide, hydrogen, methane, and carbon dioxide are shifted towards higher temperatures in the short reactor. A noticeable difference between Rensfelt's data and the 90 cm data, Figs. 3-2 and 3-4, is the decrease in carbon monoxide and the increase in hydrogen and carbon dioxide for the entire temperature range. This suggests the water-gas shift reaction which could be related to catalytic materials in the solid waste or the long residence times used by Rensfelt. Also, the relative insensitivity to temperature is to be noted in Fig. 3-2, 3-3, and 3-4.

Experiments were also run with wheat straw as the biomass feedstock. As discussed in an earlier section, wheat straw is a potential feedstock for a process upon which our research is focused. That process is the pyrolysis of agricultural nonfood products and the catalytic conversion of the resulting pyrolysis gases (primarily hydrogen, carbon monoxide, and olefins) to diesel fuel.

Results for a 30-cm reactor with particle size 150 μ m and steam-to-biomass ratio of approximately 1:1 are shown in Fig. 3-5. The most important result is that the wheat straw produces approximately half the ethylene as does the ECO-II feedstock. Possible reasons include the presence of plastics in the ECO-II as well as the fact that the ECO-II is chemically pretreated by acid hydrolysis to assist in grinding. The latter explanation seems less plausible because tests with an electrically heated platinum wire on small batches of acid-treated newsprint gave an increase in olefin yield from 10.1 to 11.2 wt % compared to nontreated newsprint. In addition, Finney (1973) has reported entrained-flow reactor results on untreated municipal waste in which half the paper fibers were removed, presumably increasing the fraction of plastics in the feedstock. Those results indicated ethylene yields of 21% by volume which is considerably higher than that reported for ECO-II yields. We, therefore, conclude that the presence of plastics is responsible for the high olefin yields reported for fast pyrolysis of municipal wastes.

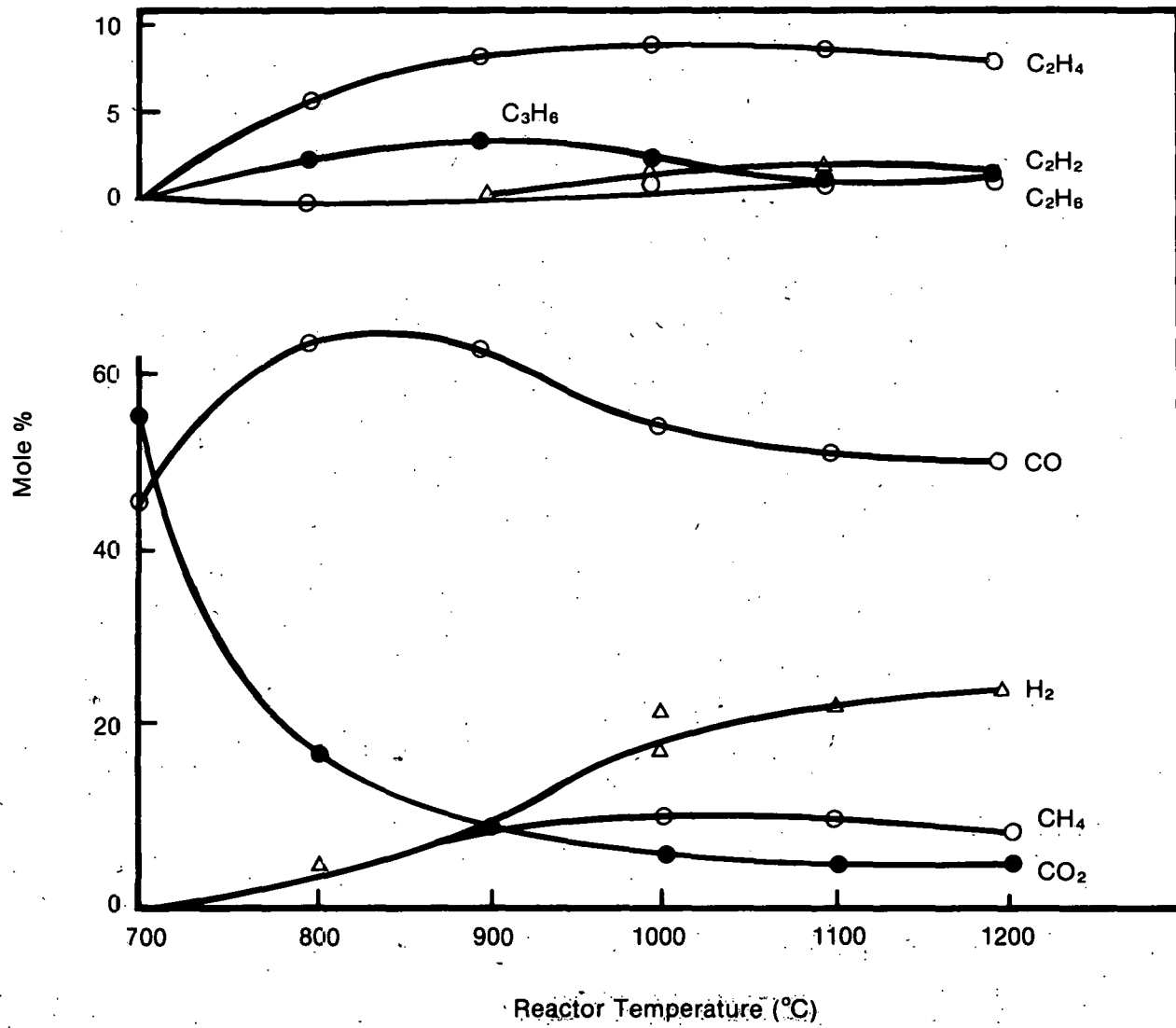


Figure 3-2. GAS COMPOSITION VERSUS REACTOR TEMPERATURE, 90-cm REACTOR, ECO-II

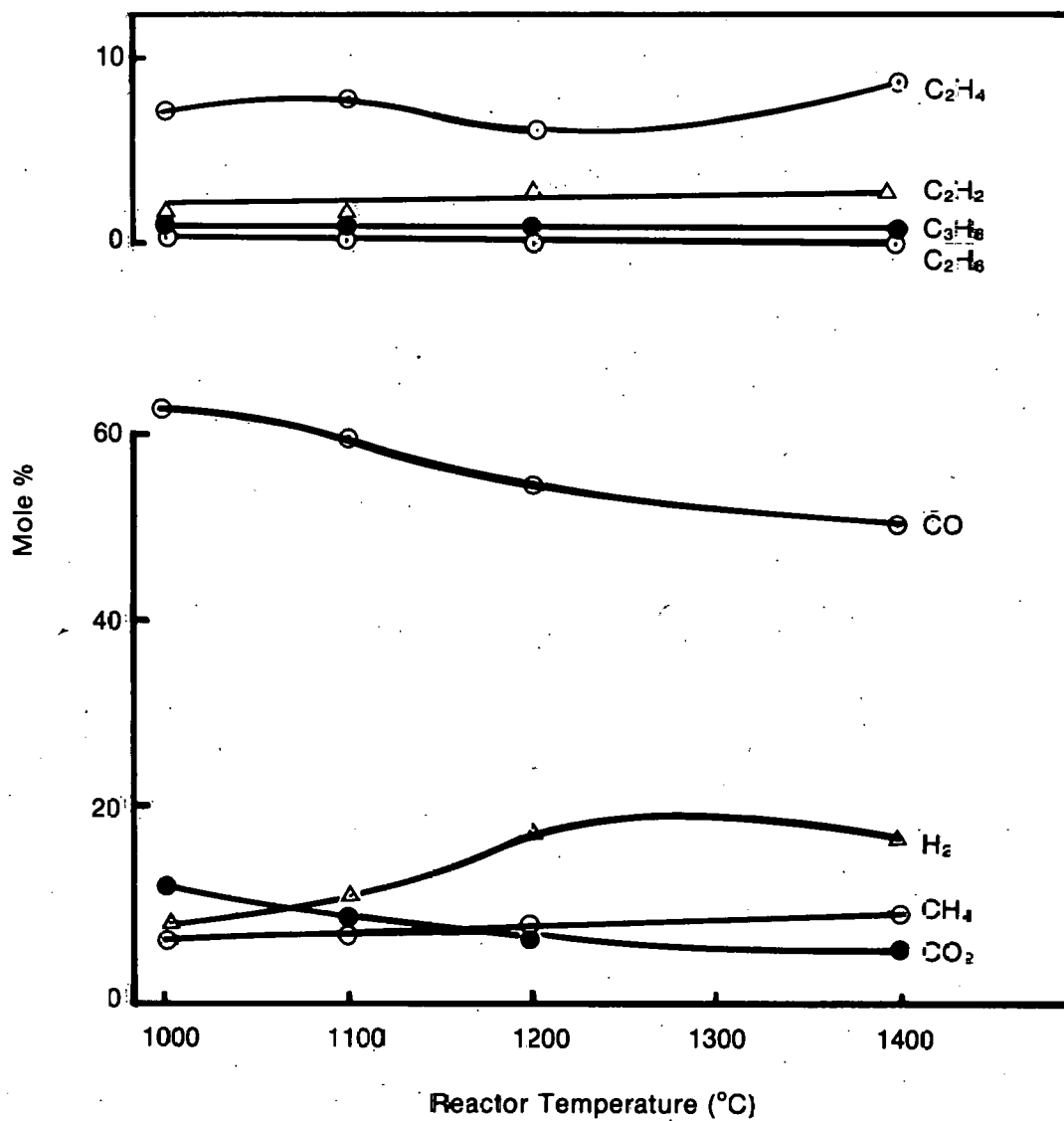


Figure 3-3. GAS COMPOSITION VERSUS REACTOR TEMPERATURE, 30-cm REACTOR, ECO-II

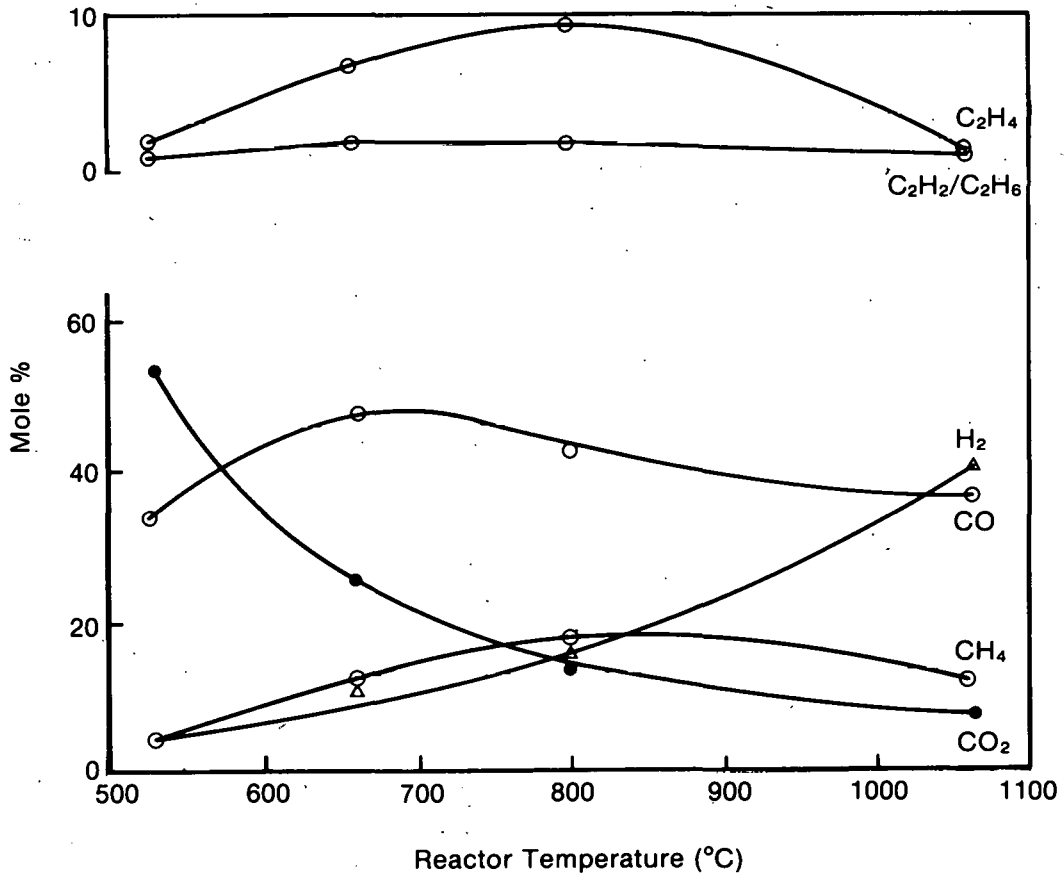


Figure 3-4. GAS COMPOSITION VERSUS REACTOR TEMPERATURE, 100-cm REACTOR, MUNICIPAL WASTE (RENSFELT)

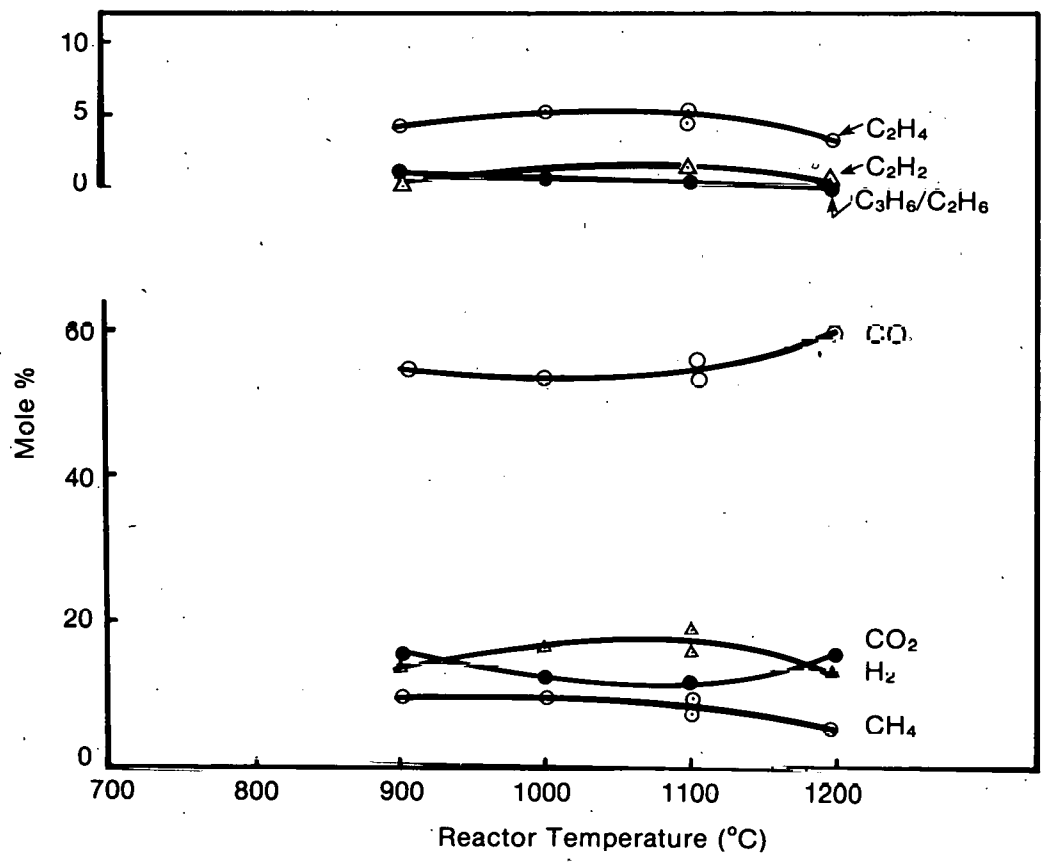


Figure 3-5. GAS COMPOSITION VERSUS REACTOR TEMPERATURE, 30-cm REACTOR, WHEAT STRAW

SECTION 4.0

PYROPROBE RESULTS

To complement our reactor experiments we have been using an electrically heated platinum wire to study fast pyrolysis of small batches of prospective feedstocks. This method is useful for screening feedstocks as well as for quickly determining operating regimes in which the reactor experiments may be interesting. The registered trade name of the device is Pyroprobe (Chemical Data Systems, Inc.) and this apparatus has been used by many researchers for similar purposes. Our work, however, indicates that some caution in interpreting results from the Pyroprobe is in order.

The Pyroprobe allows one to heat the platinum wire along a prescribed temperature-time curve. Specifically, the temperature can be programmed to increase linearly to a given temperature, and then hold that temperature for a given period of time. We used the device to heat small (200 μg) samples of fine wheat straw on a temperature ramp of $20,000^\circ\text{C/s}$ up to 1000°C and then hold the sample at that temperature for times ranging from 0.05-4.95 s.*

In Fig. 4-1 we plot the gas composition versus the time which the probe was held at 1000°C . There are clearly two regimes in operation, depending on whether the holding time is less than or greater than 0.15 s. This suggests two different mechanisms. At 0.15 s the maximum ethylene, propylene, and methane mole fractions occur. For holding times longer than this, the fraction of those components and also of carbon dioxide decreases at the same rate on the log-log scale while the hydrogen increases and carbon monoxide fraction are essentially constant. The linear decrease in the four species after 0.15 s holding time implies that either those four species cease to be generated or are generated at rates which are related in an unusual way. The most likely explanation is that the four species are produced up until 0.15 s and after that are diluted by the increasing amounts of hydrogen and carbon monoxide.

The change in mechanism may be related to poor contact of the particles with the probe. Microscopic examination of a probe after applying the particles by what seems to be the most effective method (applying a distilled water/wheat straw slurry to the element and then evaporating the water) revealed that the particles contacted the probe in only a very few isolated points. For holding times up to 0.15 sec these contact points are very rapidly heated and produce increasing quantities of olefins (and decreasing quantities of carbon dioxide, which is consistent with our low-temperature reactor experiments). During this time, it is possible that the portions of the sample not in direct contact with the probe are heated much more slowly producing char. After 0.15 s, portions of the sample in direct contact with the probe have completely pyrolyzed leaving the char to continue heating slowly and producing hydrogen and carbon monoxide. In addition, gases evolved from the particles in direct contact with the probe, could force the particles from the probe thereby greatly reducing the heating rate of the sample. A close examination of the residue left on the probe after short holding times could help verify these explanations.

*The probe is inserted into a small chamber (interface) which is plumbed into the chromatograph sample loop. The helium carrier gas purges the interface and sweeps the pyrolysis gases into the chromatograph columns.

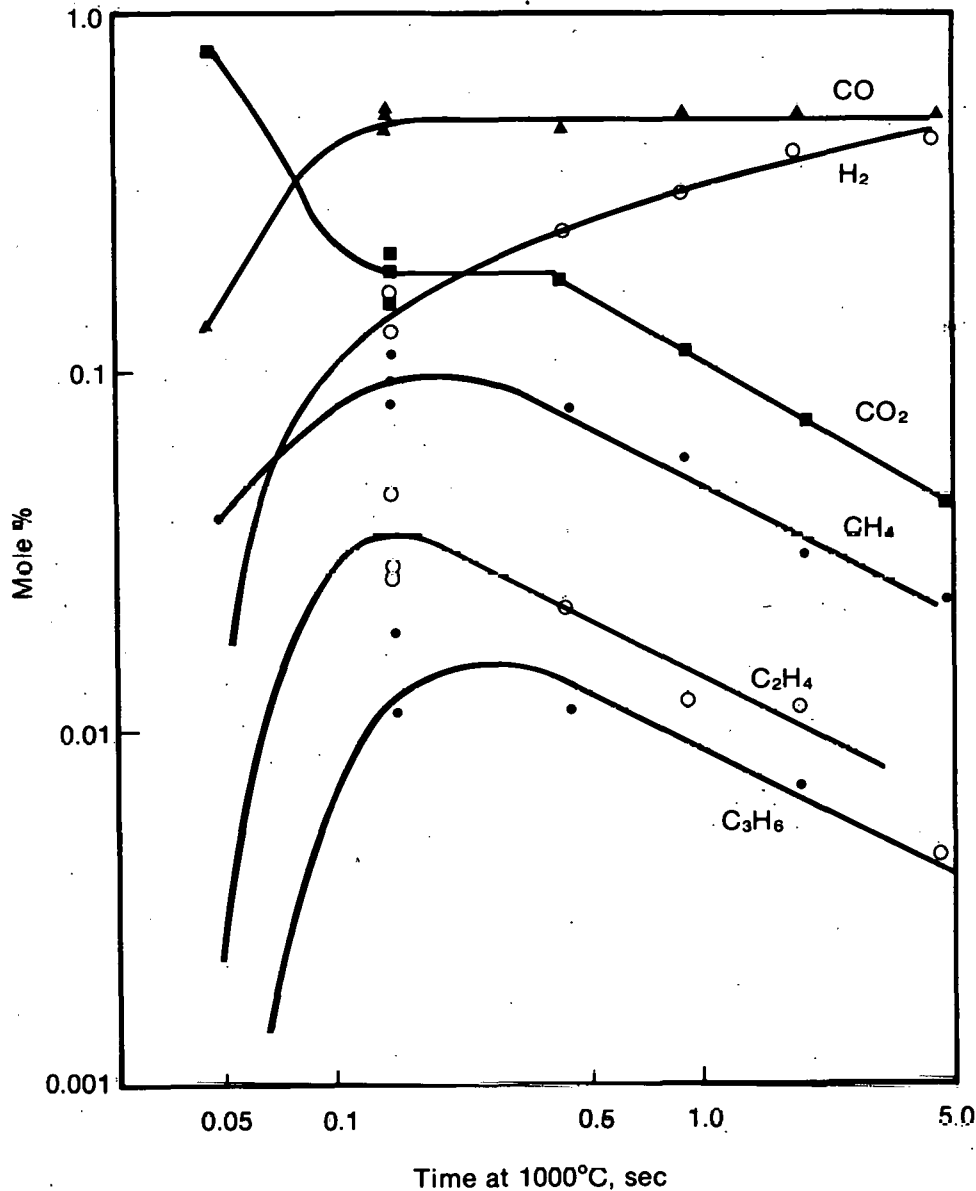


Figure 4-1. GAS COMPOSITION VERSUS HOLDING TIME (at 1000°C), WHEAT STRAW HEATED AT 2000°C/s

Comparing the gas composition after 0.15 s at 1000°C (which seems to be the gas composition most representative of the 20,000°C/s heating rate) with that for the 30-cm reactor at 1000°C, Fig. 3-5, we find they are quite similar with one exception. Pyroprobe data at these conditions is consistently lacking any meaningful quantities of acetylene, while the reactor consistently yields acetylene. A lack of acetylene implies a lower temperature reaction in the probe experiments than in the reactor experiments. Although the probe was heated at 20,000°C/s to 1000°C it appears that the particles lagged the probe temperature due to poor thermal contact with the probe. It is difficult to determine the particle temperature except indirectly by comparison with probe runs for higher final temperatures. However, an analysis of particle heating rates for the entrained reactor suggests that heating rates of 5000°C/s may be reasonable and therefore we expect that the particles heated with the probe were heated at less than 5000°C/s, and achieved temperatures less than that achieved by the particles in the 30-cm reactor.

SECTION 5.0

CONCLUSIONS

Entrained-flow reactor pyrolysis experiments on a municipal solid-waste feedstock (ECO-II) resulted in olefin yields comparable to those reported previously. However, we found trends in the data that had not been seen before: ethylene yield varies as a function of reactor temperature and residence time. The trends suggest that it may be possible to correlate gas yield with a parameter which includes temperature, reactor length and steam/biomass ratio.

We also performed experiments with wheat straw, an agricultural nonfood product. The most important result of those experiments was that olefin yields were about half that of the ECO-II feedstock. This could be due to the presence of plastics in the ECO-II feedstock.

Pyroprobe experiments with wheat straw reveal that two mechanisms affect the gas composition resulting from heating at $20,000^{\circ}\text{C/s}$ to 1000°C and then holding at 1000°C for a variable period. For periods up to 0.15 s, volume fractions of ethylene, propylene, and methane increase while that of carbon dioxide decreases. After that period the species are no longer generated and only hydrogen and carbon monoxide are generated. The change in mechanism may be related to poor thermal contact of portions of the sample with the probe. Data at 0.15 s compare quite well with the 30-cm reactor results at 1000°C except for the lack of acetylene in the pyroprobe data. This suggests that the sample was not heated as rapidly by the pyroprobe as in the entrained-flow reactor, and therefore, that some caution is justified when using the pyroprobe.

Future Work

We plan to run more experiments on wheat straw and other agricultural nonfood products in the entrained-flow pyrolysis reactor. Data from these experiments will include gas composition and percentage of gasification as a function of reactor length, reactor temperature, and steam/biomass ratio. Trends from these data will be used to develop correlations which will be useful for engineering scale-up of the reactor and to help explain pyrolysis mechanisms.

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SECTION 6.0

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15. Supplementary Notes			
16. Abstract (Limit: 200 words) <p>Pyrolysis experiments were performed using 30 and 90cm entrained-flow reactors, with steam as a carrier gas and two different feedstocks--wheat straw and powdered material derived from municipal solid waste (ECO-II™). Reactor wall temperature was varied from 700° - 1400° C. Gas composition data from the ECO-II tests were comparable to previously reported data but ethylene yield appeared to vary with reactor wall temperature and residence time. The important conclusion from the wheat straw tests is that olefin yields are about one half that obtained from ECO-II. Evidence was found that high olefin yields from ECO-II are due to the presence of plastics in the feedstock.</p> <p>Batch experiments were run on wheat straw using a Pyroprobe™. The samples were heated at a high rate (20,000° C/sec) to 1000° and held at 1000° C for a variable period of time from 0.05 - 4.95s. For times up to 0.15s volume fractions of ethylene, propylene, and methane increase while that of carbon dioxide decreases. Subsequently, only carbon monoxide and hydrogen are produced. The change may be related to poor thermal contact and suggests caution in using the Pyroprobe.</p>			
17. Document Analysis a. Descriptors Biomass ; Entrainment ; Municipal Wastes ; Pyrolysis ; Steam ; Straw ; Chemical Reactions ; Decomposition ; Energy Sources ; Renewable Energy Sources ; Thermochemical Processes ; Wastes b. Identifiers/Open-Ended Terms c. UC Categories 61d			
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