

GASIFICATION OF FEEDLOT MANURE IN A FLUIDIZED BED

The Effects of Superficial Gas Velocity and Feed Size Fraction

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INTRODUCTION

The dwindling supplies of oil and impending shortages of natural gas have made it worthwhile to consider recovering energy from solid wastes. Agricultural wastes such as feedlot manure, are one class of materials that are being investigated for possible utilization. Feedlot manure is a low sulfur material that is renewable and available in significant amounts in certain areas. Manure can be converted into useful products by anaerobic digestion, atmospheric pressure gasification or liquefaction. Of these, atmospheric pressure gasification appears to be the most economically attractive [Engler et al., (1)]. Contacting devices, such as the fixed bed, the moving bed, the entrained bed and the fluidized bed can be used for gasifying manure. From the standpoint of gas production, fluidized beds are highly desirable because of their high heat transfer characteristics and their capabilities for maintaining isothermal conditions.

A survey of the literature on the gasification of manure indicates that the available experimental data are somewhat limited. Most investigators have only examined the influence of temperature. Burton (2) carried out two experimental runs with dried cow manure in a fluidized bed reactor. The reactor used was 0.38 m (15") in diameter and employed inert matrix of sand as the bed material. Hot fluidizing gas for the reactor was generated by combusting methane or propane. The reactor operating temperatures used for the two runs reported were 1041 K and 1022 K. Smith et al., (3) published partial oxidation data obtained in a moving bed reactor using manure as the feed material. The experiments were conducted in a 0.05 m (2") diameter reactor and used recycled product gas and air as the gas medium. Data were obtained for a temperature range of 894 K to 950 K.

Bench scale operating data were obtained by Halligan et al., (4) in a 0.05 m (2") diameter reactor, which was operated in a partial combustion mode using steam and air. The reactor was externally heated with electrical heaters and the data were obtained between 977 K and 1069 K. Mikesell et al., (5) reported limited data on the flash pyrolysis of steer manure in a multiple hearth reactor. The operating temperatures for these experiments were between 873 K and 1023 K. Recently, Beck et al., (6) presented partial oxidation data on manure obtained in a pilot plant reactor. Steam and air were used as the fluidizing medium in the 450 kg/day pilot plant. The reactor used was 0.15 m (6") in diameter and had an axial temperature variation of about 500 K. The data were presented for an average temperature of about 870 K in the reactor. Howard et al., (7) have recently completed a comparative study on the gasification of a variety of biomass materials (including manure) in a 0.5 m ID fluid bed pilot plant. They examined the influence of fluidization velocity and reactor loading and gasifier performance.

To properly design a system for the gasification of manure or other biomass, it is necessary to develop systematic data base which includes the effect of operating temperature, feed size, superficial gas velocity and perhaps other variables on the gasification characteristics. These would be most useful if obtained on a pilot plant scale. The objectives of the present work were to conduct gasification experiments

with manure in a fluidized bed reactor and to assess the influence of the feed size fraction and superficial gas velocity on the following: 1) produced gas composition; 2) higher heating value of the produced gas; and 3) produced gas yield. The operating temperature was also varied in the experiments.

EXPERIMENTAL

Facilities

The pilot plant facility used for the gasification of manure is shown schematically in Figure 1. The pilot plant consisted of the following seven components: 1) the reactor, 2) a screwfeeder, 3) a cyclone separator, 4) a Venturi scrubber, 5) an afterburner, 6) a control and instrumentation panel and 7) a gas sampling train.

The reactor was constructed from heat resistant stainless steel 316 alloy. The reactor proper had an I.D. of 0.23 m (9") with an expanded freeboard of 0.41 m (16") I.D. A burner with a duty of 47.5 MJ/hr (45,000 BTU/hr) located at the bottom of the reactor (plenum) generated the gas for fluidization by the combustion of propane under starving air conditions. Water was also injected into the plenum section as necessary to maintain the temperature below 1250 K and to supply additional gas for fluidization. A sampling port was provided at the plenum section to permit monitoring of the composition of the fluidizing gas. Supplemental heat (as needed) for operation was transferred across the walls of a radiant jacket surrounding the reactor. A burner with a duty of 105.5 MJ/hr (100,000 BTU/hr) supplied heat to the jacket using natural gas as the fuel. The distributor plate for the reactor was made from a 3 mm thick 316 stainless steel plate and had 844 holes of 1.5 mm diameter. The reactor was well insulated with a minimum of 0.1 m of Kao Wool and had adequate temperature and pressure measuring elements located at various strategic points. An inert matrix composed primarily of silica sand was used as the bed material. Approximately 45 kg of sand with a mean particle size of 0.55 mm was used to give a static bed height of 0.6 m (24"). An overflow pipe for withdrawing solid samples from the bed was provided on the reactor as shown in Figure 1.

The solids to be gasified were fed into the bed through a feed pipe of 0.075 m (3") diameter, which discharged the feed just above the expanded bed surface. The feed material was delivered to the feed pipe from a sealed hopper with a variable speed screw feeder. A purge stream of about 0.36 cubic meter/hr of helium was used to maintain a positive pressure on the feed hopper as well as the feed pipe so as to prevent the backflow of off-gas into the feeder and subsequent condensation in the feeder. The off-gas from the reactor was withdrawn from the top and passed through a cyclone separator for removing the entrained solid particles which were collected in a receiver located below the cyclone. The cyclone could remove particles up to a diameter of 5 micrometers. A gas sampling point was provided at the inlet of the cyclone for monitoring the composition of the reactor off-gas. The solids free gas from the cyclone was then sent into a Venturi scrubber, which served to quench the off-gas and remove condensibles. The scrubber waste water was discharged to the sewer and the scrubbed gases were sent to an afterburner. The afterburner served as a flare stack which permitted the gas to discharge to the atmosphere after incineration.

All the temperature and flow measuring instruments and the temperature recorder for the pilot plant were mounted on a control panel. Control loops with alarms were provided to ensure safe operation. A twelve point strip chart recorder was used to monitor the temperatures at several locations, including the plenum section, the radiant section, the portion just above the distributor, the middle portion of the reactor, and the freeboard section.

A sampling train was constructed to collect samples of the plenum gas as well as the reactor off-gas. The sample stream was passed through a series of glass condensers and condensate receivers permitting the separation of condensibles from the

stream. The cooled sample gas was passed through a wet test-meter, and then through a sample bottle, and subsequently incinerated.

Feed Material Preparation

The manure used was collected from paved feedlots at Kansas State University's Beef Research Center. The manure had a moisture content of about 80% and was subsequently flash dried to reduce the moisture content to about 8%. The dry manure was sieved to obtain three size fractions, namely; -2 + 8 mesh (0.45 cm), -8 + 14 mesh (0.19 cm) and -14 + 40 mesh (0.09 cm). The ultimate analyses of the three sizes of manure are presented in Table 1.

Procedure

The reactor was initially heated to the desired operating temperature using both the plenum and radiant burners. The temperatures in various parts of the reactor were monitored to establish a stable starting condition. The propane used in the plenum burner was burnt under starving air conditions to ensure an oxygen deficient atmosphere in the reactor. Gas samples (2 or 3) were taken from the plenum section for analysis before a run was initiated. Over the course of the sampling period, condensate was collected for a measured volume of the burner gas (saturated at the metering conditions) to determine the water content of the fluidizing gas.

Manure was introduced into the reactor at a continuous prespecified rate, and the temperature profile of the reactor was closely monitored. Samples of the reactor off-gas were taken with the simultaneous collection of condensate. Run durations were 30 minutes to one hour. Feeding was then terminated and the char collected in the cyclone was weighed. Samples of the cyclone char were reserved for analysis. After the completion of each run, the char retained in the reactor was burnt with excess air and the ash produced was elutriated from the bed and collected in the cyclone. A sample of the ash generated was also reserved for analysis.

The flow rates of the propane, air and injection water were noted during each run. The solid feed rate was determined by the difference in weights of solids in the hopper before and after the experiment. For each of the runs, the gas samples were drawn after flushing the sample bottles for about five minutes. The volumetric flow of gas through the wet test meter and the pressure and the temperature of the wet test meter were noted. The condensates collected were measured volumetrically.

Chemical Analysis

Gas analysis was accomplished using a Packard Model 417 Becker Gas Chromatograph equipped with thermal conductivity detectors. The gas components of interest included H_2 , CO , CO_2 , CH_4 , C_2H_4 , C_2H_6 , C_3H_6 , C_3H_8 , H_2S , N_2 and O_2 . Column packings used were a 5A molecular sieve for the separation of H_2 , O_2 , N_2 , CO , and CH_4 , while the remaining components were separated using a column of Porapak Q with a short lead section of Porapak R to shift the retention of water. The chromatograph was operated isothermally at 80°C with helium as a carrier gas. The instrument was calibrated with purchased calibration mixtures. Solid materials were analyzed with respect to their elemental composition (C, H, N, O) using a Perkin-Model 240 Elemental Analyzer. The ash analysis was performed according to the standard ASTM procedure in a muffle furnace and the moisture content was determined by drying the samples in an oven for 3 hours at 373 K. The compositions of the solids and gases given in this study represent the average of at least two determinations.

Operating Conditions

Gasification experiments were conducted by varying the operating temperature, the feed size fraction and the gas superficial velocity. The arithmetic average

between the bed temperature and the freeboard temperature was taken as the operating temperature of the reactor. In all the experiments, the freeboard temperature was less than the bed temperature. The maximum temperature difference observed in this study was 120 K and the average temperature difference was 80 K. A summary of the operating conditions is presented in Table 2.

Data Analysis

Mass balance calculations were first performed on the plenum burner using the analysis of the dry plenum gas, the condensate collected and the flow rates of propane, air and injection water. The flow rate of the dry burner gas entering the reactor was computed by performing a nitrogen balance around the burner. An overall nitrogen balance on the reactor was then used to evaluate the dry off-gas flow rate with the aid of the off-gas analysis. For computing the amount of gas produced from the manure, it was assumed that the burner gas did not significantly take part in the reactions. The yield and the composition of the dry produced gas were computed as the difference between the dry off-gas and the dry burner gas.

From the condensate collected for a unit volume of the burner gas, the total water content of the burner gas was computed using the volumetric flow rate of the dry burner gas. Similarly, the condensate associated with the dry off-gas was computed from the volumetric flow rate of the dry off-gas and the condensate data obtained for a unit volume of the dry off-gas. The liquid produced during gasification was computed as the difference between the two after making appropriate corrections for the water of saturation of the metered gases.

To complete the overall material balance around the reactor, it was necessary to know the total amount of char produced. Since a portion of the char was retained in the bed, it was necessary to establish a procedure for evaluation of the total char generated. Attempts were made to estimate the char in the bed by performing an inert balance on the ash produced during combustion and the char. This method was not very satisfactory since substantial amounts of the ash were carried past the cyclone to the scrubber and drain. From experimental observations, it was found that for any run, the elemental analysis of the cyclone char and the char retained in the reactor agreed with each other closely. Hence, the char retained in the bed was assumed to have the same composition as that of the cyclone char. The total char produced was estimated using the ultimate analysis of char and feed coupled with an ash balance on the reactor. This procedure was subsequently checked with a pelletized feed material, whose char could be separated out from the inert solids in the bed. The check indicated that the inert balance was a satisfactory approach.

RESULTS AND DISCUSSION

Approximately 100 experiments were conducted, 45 each for the -8 + 14 mesh and -14 + 40 mesh fractions and the remainder for the -2 + 8 mesh fraction. For each run, material balance calculations were performed to evaluate the quantity and the composition of the produced gas. Material balance closure ranged from 80-115% with most runs closing to better than 90%. The higher heating value of the produced gas was calculated from its composition and the heating values of the individual components. The effects of superficial gas velocity and the feed size fraction were assessed from the results obtained.

Product Gas Composition

To examine the influence of gas superficial velocity on the concentrations of the individual components of the produced gas, data obtained at different superficial velocities were plotted against the operating temperature for a given

feed size fraction. These plots, presented in Figures 2 and 3 (for the -8 + 14 mesh and the -14 + 40 mesh sizes) showed a minimal scatter ($\pm 1\%$) indicating that for the range investigated, the gas superficial velocity did not have a discernible influence on the produced gas composition. In these two plots as well as in the subsequent ones, the actual data points are not shown for the sake of simplicity.

The effect of the size fraction used on the composition of the produced gas can be assessed by comparing Figures 2 and 3. For a given operating temperature, comparison shows that the concentrations of C_2H_6 and C_3H_8 are very close to each other for the two feed size fractions. The concentrations of CH_4 , C_2H_4 and CO_2 show similar trends in both cases. Their numerical values are in good agreement with each other up to an operating temperature of about 950 K. Beyond this temperature, the differences are more pronounced. The concentrations of H_2 and CO complement each other in the two plots. A higher value of H_2 concentration is offset by a lower value of CO concentration and vice versa. It can also be seen that the concentration of CO_2 goes through a minimum in the two figures with the numerical values for the two size fractions being distinctly different.

Figure 2 shows that for the -8 + 14 mesh size fraction, the concentration of H_2 in the produced gas varied between 19% and 35% and that of CO varied between 20% and 25%. In Figure 3 it can be seen that for the -14 + 40 mesh size fraction, the concentration of H_2 varied between 15% and 50% and that of CO between 25% and 15%. These two figures suggest that there is a distinct difference in the concentrations of CO, H_2 and CO_2 from the two size fractions. Limited data for the -2 + 8 mesh size fraction did not show an appreciable difference from the results for the -8 + 14 mesh fraction.

Heating Value

In Figure 4 the higher heating value of the gas produced at different gas superficial velocities is plotted against the operating temperature. It can be seen that the heating values go through definite maxima and then diminish. As in the case of the produced gas composition, for a given feed size fraction, the gas superficial velocity did not have a significant influence on the heating values. The deviation observed was $\pm 0.8 \text{ MJ/Nm}^3$ (+ 25 BTU/SCF).

By comparing the two curves in Figure 4, the effect of the size fraction on the heating value of the produced gas can be assessed. For the size fraction of -8 + 14 mesh, the heating value increases from 10.43 MJ/Nm^3 (280 BTU/SCF) to 19.75 MJ/Nm^3 (530 BTU/SCF) and then diminishes to 13.41 MJ/Nm^3 (360 BTU/SCF) over the temperature range studied. In the case of -14 + 40 mesh fraction, the heating value increases from 13.04 MJ/Nm^3 (350 BTU/SCF) to 18.26 MJ/Nm^3 (490 BTU/SCF) and then decreases to 12.3 MJ/Nm^3 (330 BTU/SCF). These data indicate that the feed size fraction may have a marginal influence on the heating value of the produced gas. This trend was confirmed when the data were compared with a limited number of data obtained for a -2 + 8 mesh fraction of manure as shown in Figure 4. A comparison of the heating value curves for the three size fractions indicates that the smaller the size, the lower the heating value of the produced gas at temperatures above 900 K. The peak of the heating value curve shows a shift to the right as the size fraction becomes larger. Also, as size increases, the peaks become narrower.

Produced Gas Yield

Figure 5 presents plots of the yield of dry produced gas (on a dry ash free basis) versus temperature for the different size fractions. The data points for a given size fraction showed a fair amount of scatter. The scatter was such that bands of $\pm 0.25 \text{ Nm}^3/\text{kg}$ about the lines shown in Figure 5 were needed to contain the data for

a given size fraction. There were no discernible trends in the data to suggest that superficial velocity variations were responsible for the scatter observed. The average yield of dry produced gas ranges from 0.13 Nm³/kg (2.1 SCF/lb) at 820 K to 0.86 Nm³/kg (13.8 SCF/lb) at 1020 K for the -14 + 40 mesh fraction. For the -8 + 14 fraction the average yield ranges from 0.04 Nm³/kg (0.6 SCF/lb) at 820 K to 0.72 Nm³/kg (11.5 SCF/lb) at 1020 K. A limited amount of data for the -2 + 8 size fraction are also presented in Figure 5. The comparison shows a definite tendency for higher gas yields with smaller feed size fraction.

A simple conceptual model for the gasification of manure can be envisioned to consist of the following steps: 1) Devolatilization of the solid to form char and volatile matter; 2) Thermal cracking of heavy volatiles to produce light components and char (carbon deposition) and gas phase water-gas and steam-hydrocarbon reactions. The yield of total volatiles in the first step will dictate the level of gas yield that can be obtained from the solid feed. The extent of thermal cracking and other gas phase reactions of the volatiles is determined by their time-temperature history. These reactions will establish the final ratio of gas to liquid and the gas composition.

Thermogravimetric studies on manure have indicated that the devolatilization step starts around 420 K and is complete around 770 K [Howell, (8)]. Statistical analysis of additional data taken in this laboratory indicate that the heating rate employed (40 K/min to 160 K/min) has no effect on the devolatilization characteristics. Antal's work (10) on manure indicates a slight dependence on heating rate (5 K/min to 140 K/min) but this was not examined to determine if it was statistically significant. Anthony and Howard (9), in their work with coal, have argued that high heating rates (10,000 K/sec) give a greater extent of devolatilization than can be obtained with normal TGA heating rates.

Since the rate of heat transfer is very high in the fluid bed (1000 K/sec) and normal operating temperatures are well above those for completion of devolatilization, it can be assumed that the devolatilization step takes place instantaneously. Further more, for the range of temperature employed, it is anticipated that the variation in operating temperature has little effect on the extent of devolatilization. In this work comparisons are made at a given operating temperature and even if a temperature dependence of the devolatilization did exist, the phenomenon would not be a variable that influenced the comparison. Consequently the devolatilization phenomenon can be ruled out as a cause for the observed variations in gas yield for a given feed size.

Antal (10) conducted studies of the vapor phase cracking reactions with volatiles produced from cellulose under conditions where the devolatilization phenoma was held constant. He found that at a given temperature, the amount of each component in the produced gas was affected by the residence time. His results indicated that for residence times up to about 5 seconds, the yield of the components such as CH₄, C₂H₆, C₂H₄ and C₃H₆ increased dramatically and beyond five seconds the effect was much less pronounced. He also found that the amount of each component increased with temperature and in the case of both C₃H₆ and C₂H₆ the amounts of each passed through maxima as temperature increased.

In the present work, the residence time of the gas in the reactor was calculated to be approximately six seconds. The variation in this value over the experimental range was about ± 1 second. Since this value is more than five seconds and the variation observed is not extensive, it can be expected that the gas residence time does not have a significant effect on the data. This is corroborated by the experimental observation that the superficial velocity of the gas, which is related to the residence time, did not have a significant influence on the gas composition and heating value for a given feed size fraction and a given gasification temperature. However, the yields obtained with a given feed size fraction and temperature showed a significant scatter

which is far beyond the bounds of variations that can be expected on the basis of the variations in the time-temperature history that the produced gases experience.

One plausible explanation for the observed behavior might lie in possible variations in the feed make-up as a consequence of segregation effects between batches. This is supported by the observation made during the test program that the gas yield data for a given batch of manure were consistent but varied from batch to batch for a given feed size and operating temperature. An examination of the elemental analysis of the different feed batches did not indicate significant variations in elemental composition. Since manure consisted of a mixture of stalks, hulls and other plant materials, it was next decided to examine possible variations in the cellulose content of these components. Whistler and Smart (11) indicate that a considerable variation in cellulose content exists for different parts of a plant as well as between different types of vegetation. For example, leaves contain 10-20% cellulose, stalks, 40-50%, hulls, 35% and cobs, 40% cellulose. Consequently segregation phenomena between batches could give rise to feeds with different cellulose content.

The influence of cellulose content on the devolatilization characteristics of biomass materials was then examined. Howard et al. (7) reported on the maximum oil yield obtainable from different biomass materials. The maximum oil yield can be related to the extent of devolatilization that will take place for a given material. In their work with paper, sawdust and mixtures of the two, it was observed that the maximum oil yield increased in the order sawdust, mixture, paper. The cellulose content increases in the same order. Their study does not relate this observation to the gas yield, unfortunately.

In order to examine this dependence further, limited data on the cellulose content, TGA analysis and gas yields for cellulose, paper hardwood, softwood, manure and coal were compared. Table 3 presents the summary of the TGA results obtained by Antal (10) for cellulose, paper and wood and by Howell (8) for manure and coal. The cellulose content as well as the relative gas yield for some of these materials are also presented for comparison. The relative gas yield is for 970 K with the result for cellulose from Antal (10) and the remaining values from this laboratory. As these limited data indicate, it appears that the TGA results, gas yields and cellulose content show the same trends implying that increasing cellulose content may correlate with increasing devolatilization. This possible correlation needs to be examined further.

It is quite possible in this work, that between batches, the cellulose content of the manure feed could have been different due to segregation. This difference could very well be responsible for the scatter observed in the produced gas yield for a given feed size fraction. In view of this, caution should be exercised in interpreting the influence of particle size on the gasification characteristics of biomass.

For the different feed size fractions, variations in the material make-up were evident. The -2 + 8 mesh size fraction consisted of hulls and undigested grain. The -8 + 14 mesh size fraction was spherical in shape and had a small amount of undigested grain, whereas the size fraction of -14 + 40 mesh was comprised of fine strands of stalks.

The differences in heating value and yield observed for different size fractions could well be due to variations in material make-up alone but it cannot be ruled out that particle size may also have some influence. Maa and Bailey (12) in their study on cellulosic materials, theorized that for particle sizes less than 0.2 cm in diameter, pyrolysis is reaction controlled and the particle size has no influence. In the present study the size fraction -14 + 40 mesh (0.09 cm) falls below this value, the size fraction -8 + 14 mesh (0.19 cm) is marginally below, while the size fraction -2 + 8 mesh (0.45 cm) is above the 0.2 cm size stipulated by Maa and Bailey.

Consequently for this study size effects should not be important for the smallest size fraction but may be intruding for the other two sizes, especially the largest size.

CONCLUSIONS

Gasification studies were conducted with different size fractions of manure particles in a fluidized bed reactor. The effects of gas superficial velocity and feed size fraction on the gasification were studied at different operating temperatures. Superficial gas velocity did not appear to have a significant influence on the composition and heating value of the produced gas. The feed size fraction did have a definite influence on the composition, heating value and yield of the produced gas. The observations indicate that the yield increases and the heating value decreases as the size fraction becomes smaller. In the conduct of the experiments considerable scatter was observed in the gas yield obtained with different batches of feed for a given operating condition. A possible explanation for this behavior is offered which suggests that segregation phenomenon between batches of feed and subsequent variations in the cellulose content of the batch may be primary factors influencing the observed scatter. The apparent correlation between the cellulose content and the gas yield needs further examination.

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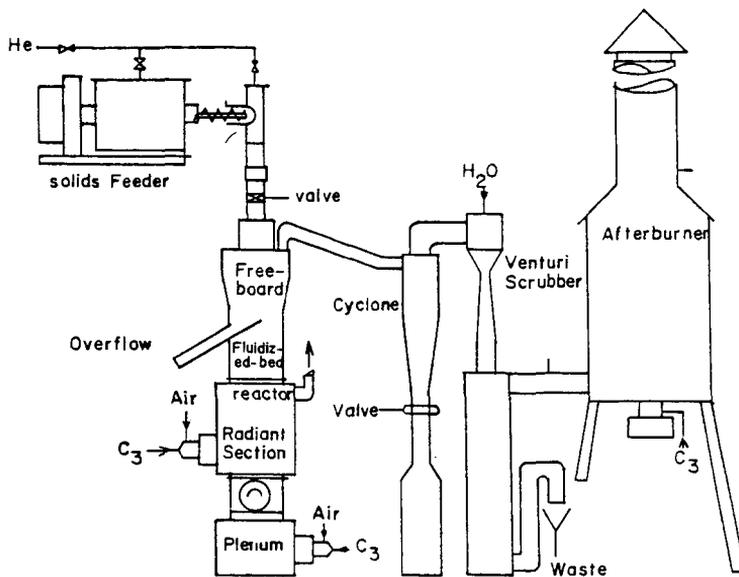


Figure 1. Flow scheme of the Pilot plant.

Table 1. Elemental Analyses of the Feed

| | -2 + 8 mesh (wt %) | -8 + 14 mesh (wt %) | -14 + 40 mesh (wt %) |
|-------------------|-----------------------|------------------------|-------------------------|
| C | 43.1 | 38.9 | 41.1 |
| H | 5.8 | 5.4 | 5.2 |
| N | 3.0 | 3.1 | 3.3 |
| O (by difference) | 26.5 | 30.4 | 30.0 |
| Moisture | 4.8 | 9.4 | 6.6 |
| Ash | 16.8 | 12.8 | 13.8 |

Table 2. Summary of Operating Conditions

| Feed Size Fraction | -2 + 8 Mesh | -8 + 14 Mesh | -14 + 40 Mesh |
|----------------------------------|--------------|--------------|---------------|
| Feed Rate (kg/hr) | 11.0 to 17.2 | 11.9 to 30.2 | 5.1 to 31.8 |
| Reactor Temperature (K) | 900 to 980 | 800 to 1040 | 800 to 1040 |
| Superficial Gas Velocity (m/sec) | 0.31 to 0.37 | 0.33 to 0.45 | 0.33 to 0.45 |
| Injection Water Rate (kg/hr) | 2.0 to 2.5 | 2.0 to 3.5 | 2.0 to 3.5 |

Table 3. Devolatilization Characteristics of Different Materials

| Materials | Total Devolatilization (weight %) | Relative gas yield | Cellulose content (weight %) |
|----------------------|--------------------------------------|-----------------------|------------------------------------|
| Cellulose | 90 | 11 | 100 |
| Paper | 85 | - | |
| Cherry (Hardwood) | 80 | - | |
| Pine (Softwood) | 70 | - | 58 |
| Cane (Sorghum) | - | 7 | 35-50 |
| Manure | 55-60 | 5 | |
| Coal | 30 | 3 | 0 |

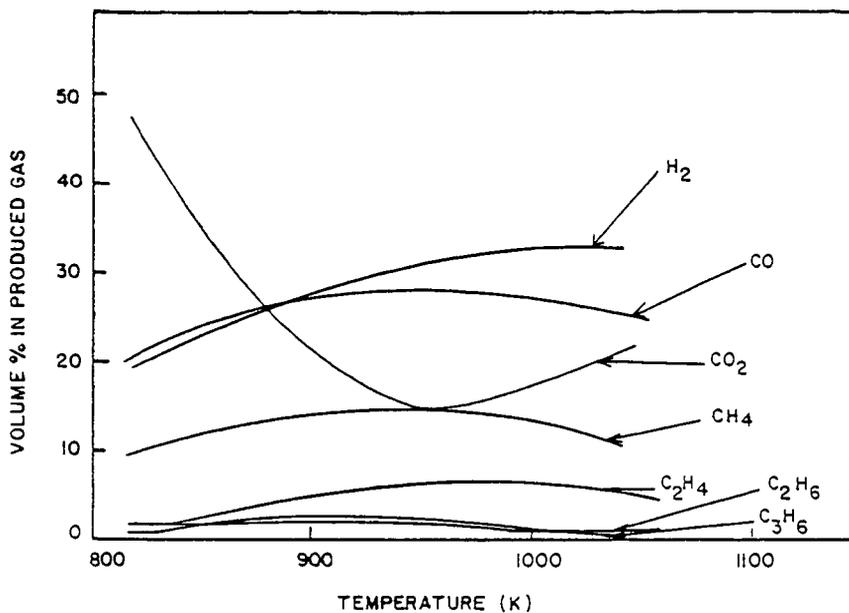


Figure 2. Produced gas composition versus temperature for -8 + 14 mesh size.

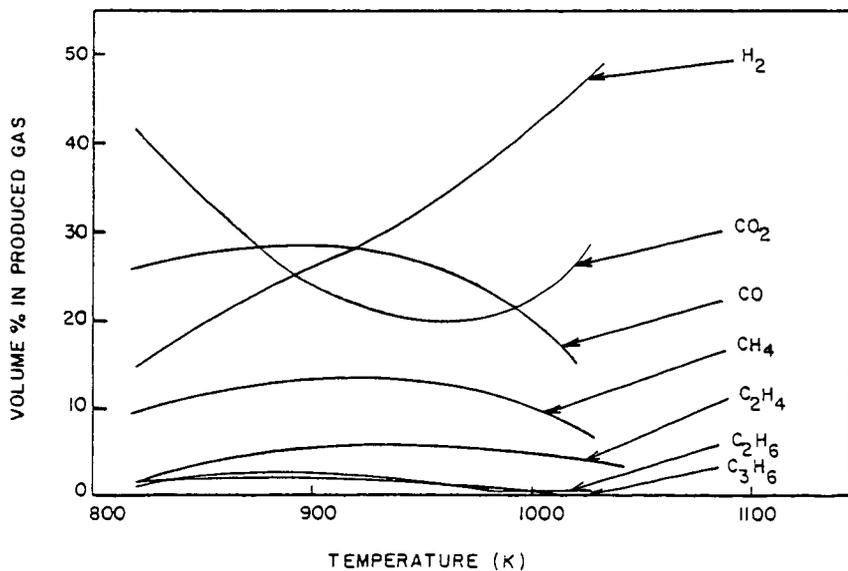


Figure 3. Produced gas composition versus temperature for -14 + 40 mesh size.

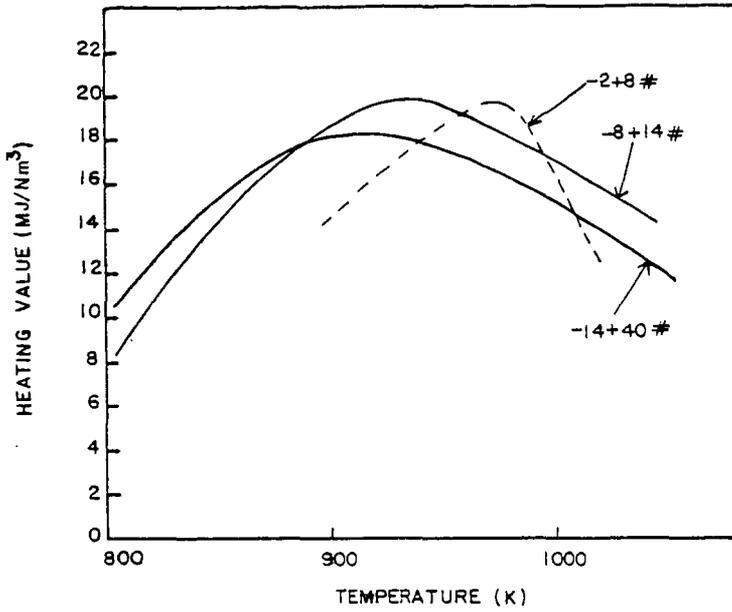


Figure 4. Gas higher heating value versus temperature.

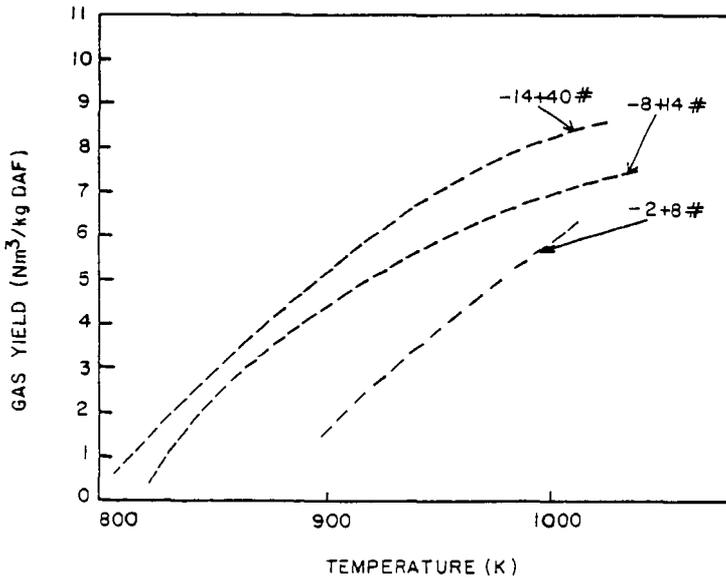


Figure 5. Gas yield versus temperature.