An Improved Pyrolyzer for Solid Waste Resource Recovery in Space

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ABSTRACT

Pyrolysis processing is one of several options for solid waste resource recovery in space. It has the advantage of being relatively simple and adaptable to a wide variety of feedstocks and it can produce several usable products from typical waste streams. The overall objective of this study was to produce a prototype mixed solid waste pyrolyzer for spacecraft applications. A two-stage reactor system was developed which can process a maximum of about 0.5 kg of waste per cycle. The reactor includes a pyrolysis chamber where the waste is heated to temperatures above 600 °C for primary pyrolysis. The volatile products (liquids, gases) are transported by a N_2 purge gas to a second chamber which contains a catalyst bed for cracking the tars at temperatures of about 1000-1100 °C. The tars are cracked into carbon and additional gases. Most of the deposited carbon is subsequently gasified by oxygenated volatiles (CO_2, H_2O) from the first stage. In a final step, the temperature of the first stage can be raised and the purge gas switched from N_2 to CO_2 and/or O_2 in order to gasify the remaining char in the first stage and the remaining carbon deposits in the second stage. Alternatively, the char can be removed from the first stage and saved as a future source of CO_2 or partially gasified to make activated carbon. This paper describes several improvements that were made in the original (First Generation) prototype pyrolyzer including: 1) replacement of stainless steel flanges with machineable ceramic in order to reduce weight; 2) construction of a new sample holder in order to make sample insertion and removal easier and sample heat-up more uniform; 3) replacement of a stainless steel outer shell with a double-wall quartz cylinder in order to significantly reduce weight and heat losses. In addition, experimental results are included for wheat straw and chicken manure feedstocks, primarily from the First Generation prototype.

INTRODUCTION

A key element of a Controlled Ecological Life Support System (CELSS) is a means for solid waste resource recovery. Solid wastes in a spacecraft environment will include inedible plant biomass (IPB), paper, plastic, cardboard, waste water concentrates, urine concentrates, feces, etc. It would be desirable to recover usable constituents such as carbon, CO_2, H_2O, hydrogen, nitrogen, nitrogen compounds, and solid inorganics. Any unusable byproducts should be chemically and biologically stable and require minimal amounts of storage volume. Many different processes have been considered for dealing with these wastes: incineration, aerobic and anaerobic biodigestion, wet oxidation, supercritical water oxidation, steam reforming, electrochemical oxidation and catalytic oxidation [1-13]. However, some of these approaches have disadvantages which have prevented their adoption. For example, incineration utilizes a valuable resource, oxygen, and produces undesirable byproducts such as oxides of sulfur and nitrogen. Incineration also will immediately convert all of the waste carbon to CO_2, which may require storing excess CO_2. Supercritical water oxidation requires the use of thick-walled high pressure equipment which is expensive to transport into space.

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"Pyrolysis," in the context of this paper, is defined as thermal decomposition in an oxygen-free environment. Primary pyrolysis reactions are those which occur in the initial stages of thermal decomposition, while secondary pyrolysis reactions are those which occur upon further heat treatment. A pyrolysis-based process has several advantages when compared to other possible approaches for solid waste resource recovery: 1) it can be used for most types of solid products and can be more easily adapted to changes in feedstock composition than alternative approaches; 2) the technology is relatively simple and can be made compact and lightweight and thus is amenable to spacecraft operations; 3) it can be conducted as a batch, low pressure process, with minimal requirements for feedstock preprocessing; 4) it can produce several useful products from solid waste streams (e.g., CO_2, H_2O, H_2, NH_3, CH_4, etc.); 5) the technology can be designed to produce minimal amounts of unusable byproducts; 6) it can produce potentially valuable chemicals and chemical feedstocks; e.g., nitrogen-rich compounds for fertilizers, monomers, hydrocarbons); 7) pyrolysis will significantly reduce the storage volume of the waste materials while important elements such as carbon and nitrogen can be efficiently stored in the form of pyrolysis char and later recovered by gasification or incineration when needed. In addition to being used as the primary waste treatment method, pyrolysis can also be used as a pretreatment for more conventional techniques, such as incineration or gasification. A summary of the proposed pyrolysis processing scheme is included in Reference 14. Additional information on the application of pyrolysis processing to space waste can be found in a recent paper by Green and co-workers [15] at the University of Florida.

The primary disadvantages of pyrolysis processing are: 1) the product stream is more complex than for many of the alternative treatments; 2) the product gases cannot be vented directly in the cabin without further treatment because of the high CO and H_2 concentrations. The former issue is a feature of pyrolysis processing (and also a potential benefit, as discussed above). The latter issue can be addressed by utilization of a water-gas shift reactor to remove CO or by introducing the product gases into an incinerator or high temperature fuel cell.

An earlier paper included the design and operating procedures for the First Generation prototype [14]. The current paper describes the evolution of the prototype pyrolyzer from the initial design (First Generation) to Second and Third Generation units. It also includes experimental results from the First and Second Generation prototypes.

EXPERIMENTAL METHODS

SAMPLE SELECTION

In previous work at Advanced Fuel Research, Inc. (AFR) [16] and Hamilton Sundstrand Space Systems International (HSSSI) [11], a model waste feedstock was used, the so-called "Referee Mix." This composite mixture consisted of 10 wt. % polyethylene, 15% urea, 25% cellulose, 25% wheat straw, 10% Gerepon TC-42 (space soap) and 5% methionine. The materials that were obtained and the elemental compositions of each are given in Reference 16. For the development of the prototype pyrolyzer, the primary focus has been on wheat straw as the test waste stream, since it is available at a relatively low cost and its elemental composition is similar to the average elemental composition of the composite mixture used previously. Three different samples of wheat straw have been used to date. The elemental composition of each is provided in Table 1. The moisture content of each is about 5-7 wt.% (as-received basis).

The possibility of obtaining hydroponic biomass as being a more realistic case was also considered. However, for the same reasons discussed in Reference 17, this was not done, due to cost and availability factors, and the fact that the mineral content is so dependent on the hydroponic environment. In addition to wheat straw, which has been commonly used by NASA as an analog for inedible biomass, the use of human waste as a feedstock was also considered. However, because of the special handling requirements, it was decided to use poultry manure (litter) instead [18]. It can also be seen from Table 1 that the elemental compositions of the human feces and chicken manure and the wheat straw samples are similar for the major components (C,H,O) and differ primarily in the minor components (N,S) and in the ash content and ash composition (not shown).

In earlier experiments involving wheat straw pyrolysis, a finely ground "Danish" wheat straw sample was used [14]. Since that material was consumed and, a new supply was not available, a simple laboratory scale facility of chopping and grinding raw wheat straw was established. The wheat straw (i.e., straw bales) was purchased from a local farm and then decreased in size in a stepwise manner. First, it was cut by hand to 3-5 inch long pieces, and then further decreased in size using a common household food processor. However, the particle size of the wheat straw prepared this way was significantly larger relative to the earlier samples of Danish wheat straw. The two materials are shown in Figure 1.

Table 1 - Composition of the various waste samples (wt. %, dry ash free basis)

Species	C	H	O	N	S	Ash[c]
NIST wheat straw	48.0	6.2	44.9	0.68	0.21	9.9
Danish wheat straw	50.4	6.0	42.5	0.86	0.25	7.9
Local wheat straw	49.0	6.1	42.9	1.8	0.2	6.0
Human Feces[a]	49.1	7.2	NA	2.7	NA	12.5[d]
Poultry litter[b]	47.4	6.5	39.5	5.6	1.0	22.1

Notes: NA = not available; a = reported by Ref. 17; b=reported by Ref. 18; c=dry basis; d=average

Figure 1. Comparison of locally prepared wheat straw sample (left) with Danish wheat straw sample (right).

REACTOR SYSTEM

A schematic of the original (First Generation) two-stage pyrolysis reactor system is shown in Figure 2. The system was designed in order to incorporate the pyrolysis, tar cracking, and gasification steps into a single reactor unit with two chambers [14,19]. The outer closure of the First Generation Unit (FGU) was a stainless steel tube with flanges on both sides. It was manufactured by welding commercially available 8 inch half nipples and flanges (Huntington Lab. Inc.) onto both ends of an 8 inch stainless steel tube. The copper gasket seals between the reactor body and flanges allowed for about a 450 °C maximum shell temperature. The thermal insulation and electric heaters were placed inside the tube. This allowed for operation of the reactor at high (up to 1100-1200 °C) temperatures without the necessity to excessively increase the size and mass.

The inner volume was divided into two chambers, and the temperature of each was regulated independently. The right chamber was partially filled with silica xerogel and functions to completely break down the tar produced in the pyrolysis chamber to elemental carbon and light gases. In a previous project on diesel fuel pyrolysis, xerogel was found to be an exceptionally good catalyst for cracking carbonaceous materials to carbon as well as catalyzing high temperature gasification of the carbon deposited on the xerogel surface [20].

During the initial processing step, the first stage is the primary pyrolysis zone, while the second stage is the secondary pyrolysis zone. The tar formed in the pyrolysis chamber is carried downstream with an inert (nitrogen) gas. During the second processing step, the purge gas is switched from N_2 to CO_2 and gasification of the char can occur in the first stage (if the temperature is raised sufficiently) while gasification of the carbon deposits will occur in the second stage.

A slip stream of the exhaust gas was created with a teflon piston/cylinder pump. The flow rate of this stream was regulated with a Hewlett-Packard DC power supply and directed through an infrared (FT-IR) spectrometer for analysis. The FT-IR spectrometer was an Online Technologies, Inc. Model 2010 mid-IR spectrometer equipped with a multi-pass cell, allowing for continuous monitoring of a variety of gases.

The temperature of the two reactor chambers was controlled independently using two thermocouples, solid state relays and two AC regulated 3 kW power supplies. The steady state power consumption was about 600 watts, which increased 15%-20% during the active pyrolysis period.

The entire control and data collection operation was facilitated through one PC running a National Instruments LabView™ program written for this particular apparatus. The data collection (except for the FT-IR data) and control functions were interfaced to a National Instruments 6023 board and the serial port of the PC.
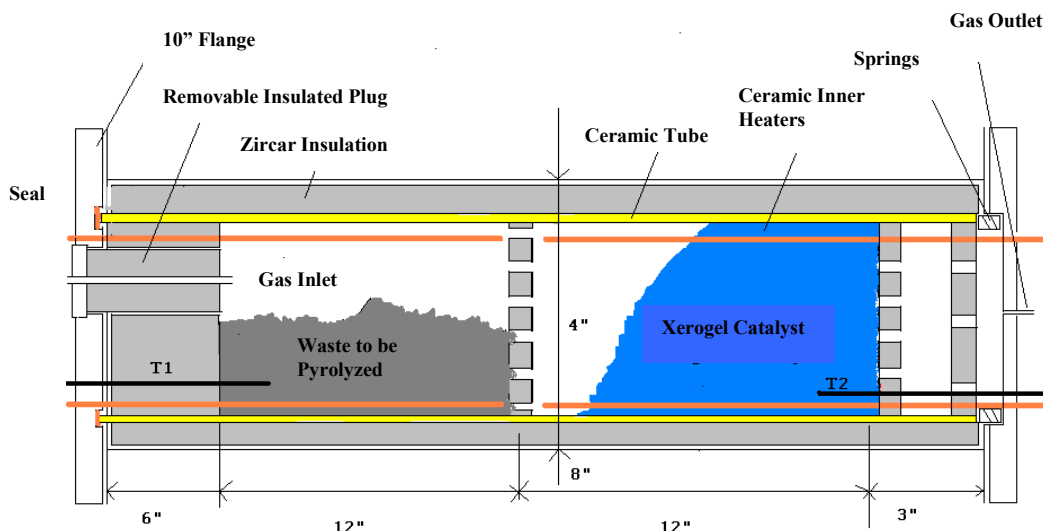
Figure 2. Schematic of First Generation two-stage pyrolysis reactor system.

Additional details of the FGU design and operating procedures can be found elsewhere [14]. The experimental results presented in the next section are from the FGU unless otherwise noted. This operating experience led to several design changes which are incorporated into the Second Generation Unit (SGU) and the Third Generation Unit (TGU).

RESULTS AND DISCUSSIONS

Approximately 0.3 to 0.4 kg of sample is placed in the reactor and the system is constantly purged with 800 cc/min N_2. First, the secondary pyrolysis chamber is heated up to about 1100 °C. Next, the temperature of the primary pyrolysis chamber is raised at approximately 5-7 °C/min to 600 °C, with about a 10 minute hold period, while the volume and composition of the product gases is constantly monitored. Selected experiments were done at a slightly higher heating rate (8-9 °C/min). The following gas concentrations are routinely monitored: H_2O, CO, CO_2, CH_4, C_2H_4, NO, NO_2, SO_2, H_2S, NH_3, CH_3OH, C_6H_6, $C_6H_5CH_3$. The main products were CO_2, CO, CH_4, H_2O and some aromatic products (C_6H_6, $C_6H_5CH_3$) while the other gases appeared only in trace amounts. Some H_2O is produced during pyrolysis and it is also present in the samples as moisture.

Figure 3 shows results for the time-temperature history and the total gas evolution rate for a typical experiment in the FGU. Figure 4 shows the concentrations of CO, CO_2, CH_4 as well as H_2 in the exhaust gases for an experiment done under similar conditions, but in the SGU. The H_2 evolution was estimated by subtracting the three main products and the carrier N_2 flow from the measured volumetric flow at the reactor outlet. The overall material balances for both the FGU and SGU were typically closed to within 90-95%.

After the pyrolysis reaction step (~120 minutes), the purge gas was usually switched to CO_2 to gasify the carbon deposited in the second chamber. In many of the later experiments, the primary char sample was removed from the first stage and the carbon deposited in the second stage was burned off with O_2. It appears that O_2 is more effective in restoring the catalyst activity (see below).

Most of the experiments in the FGU and SGU were run with pure wheat straw (without additives). A comparison of the results for finely ground ("Danish") and coarse ("Local") wheat straw at approximately the same heating rate (8-9 °C/min) is shown in Table 2 for runs #16 and #28, respectively. Significant differences were observed in the product yields between the two samples, which may be due to the particle size differences. The coarse sample yielded lower deposited (stage 2) carbon and water, together with very high ethylene yields. However, the ethylene yield decreased, if a slower heating rate (5-7 °C/min) was applied (see run #'s 29,30,31).

It was observed that gas evolution began at lower temperatures with the coarse straw than with the finally divided wheat straw [19]. This indicated the presence of "hot spots" in the reactor and the sample holder redesign (see Figure 5) was done partly for this reason, as discussed below.

The deposited carbon onto the stage 2 catalyst surface was burned with oxygen at 750 °C in the experiments before run #28. In the runs #28-#30 the carbon was burned at 1050 °C. The data in Table 2 shows that treatment of the catalyst with oxygen at 1050 °C caused stepwise (run by run) improvement in increasing hydrogen yield and decreasing carbon deposition. All of the results reported in Table 2 are from the FGU, except

Table 2 –Results from NASA Two-Stage Pyrolyzer Experiments with Wheat Straw samples (wt.%, as-received basis)

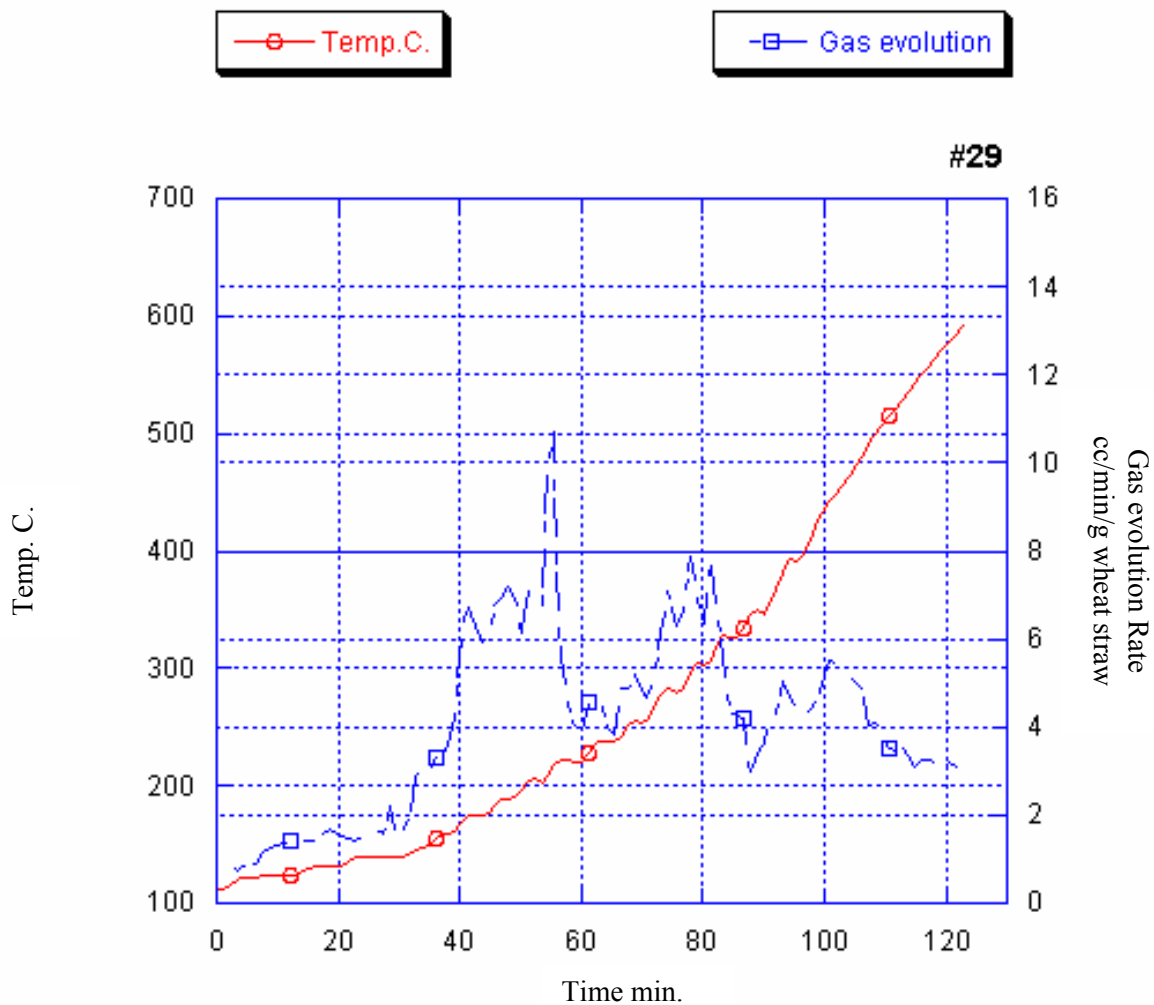| Post Pyrolyzer Temperature	1100 °C	1100 °C	1100 °C	1100 °C	1100 °C	1100 °C
Run #	16	28	29	30	31	33
Type	Fine	Coarse	Coarse	Coarse	Coarse	Coarse
Char	30.2	30.9	30.9	30.5	30.4	31.9
H_2O	24.9	5.7	9.8	7.8	9.6	9.5
Carbon	7.6	1.9	4.8	3.1	1.9	<1.0
Trap & Filter	10.5	13.2	6.9	2.8	93.9	~0
Total Gases	34.4	48.5	47.5	54.6	53.8	56.1
H_2	1.9	2.1	1.6	2.3	2.8	5.0
C_2H_4	0.1	12.0	0.2	0.1	0.2	0.9
CH_4	2.2	2.1	3.4	3.1	3.5	4.2
CO_2	17.2	23.2	24.7	26.7	32.2	30.1
CO	13.0	9.2	17.6	22.3	15.1	15.9

Figure 3. Experimental results for time-temperature history and total gas evolution for run in FGU with unground wheat straw sample (#29).
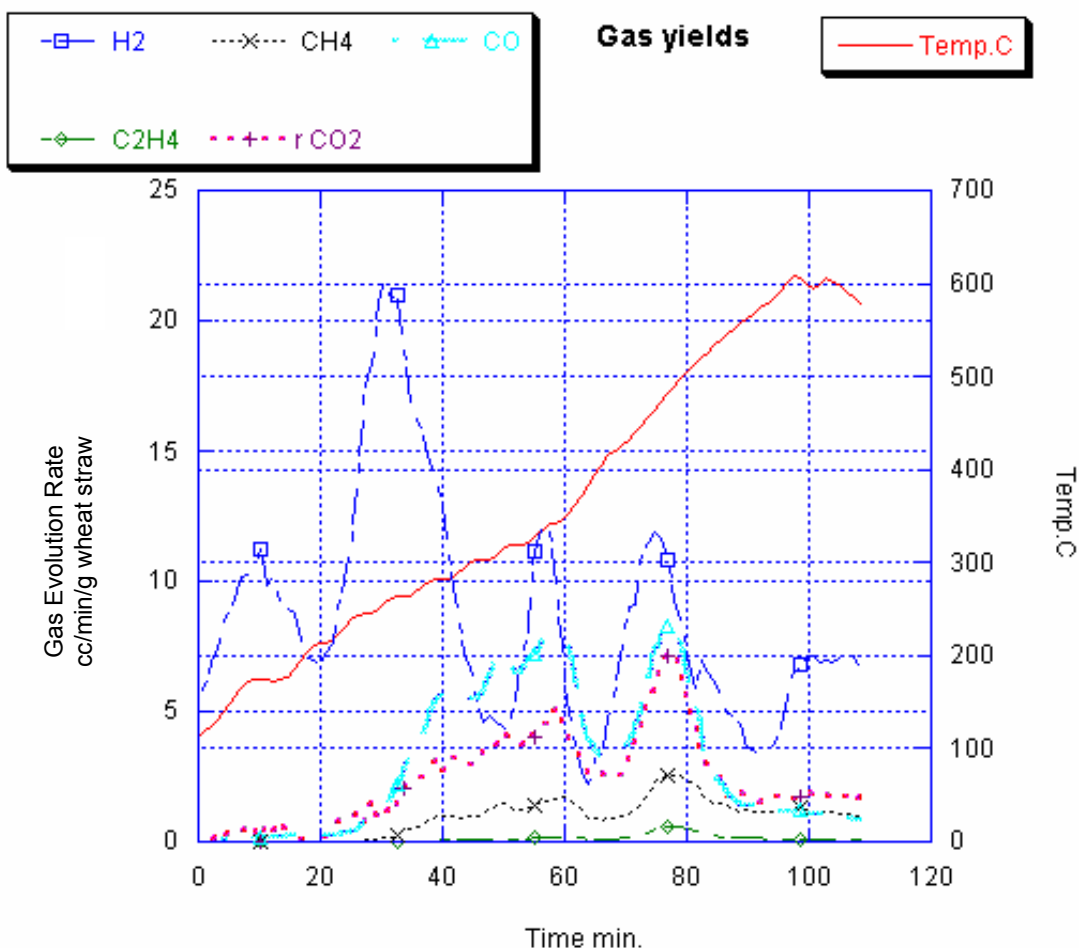
Figure 4. Experimental results for time-temperature history and individual gas evolution rates for run done in SGU with unground wheat straw sample (#33).

for #33 (see below). In run #30, the reactor outlet became partially plugged at the beginning of the run, resulting in an increase of the pressure to 20 psig. While this did not significantly affect the overall yields, it influenced the CO_2/CO ratio.

A set of experiments was done under similar conditions with the chicken manure sample in the FGU and these results are shown in Table 3. The yields for char were about 10 wt. % higher than for wheat straw, which is consistent with the higher amount of ash in the starting material (see Table 1). The yields of H_2 are somewhat lower than from the wheat straw sample, which is probably due to the fact that much of the hydrogen in the starting sample ends up as NH_3 in the product gas. The yields of CH_4, CO, and CO_2 were generally similar in either case, although the yields dropped significantly when the second stage temperature was lowered from 1100 °C to 1050 ° (see Table 3, run #27).

The above observations indicated two potential problem areas with the First Generation prototype: 1) the presence of hot spots in the pyrolyzer chamber and 2) the tendency of material deposition to cause plugging

at the reactor outlet. The material deposition occurred on the cold metal effluent tube located immediately after the metal flange. It was also found that, by heating this outlet tube, the accumulation of organic material could be avoided. However, this is not a permanent solution since it exposes the seals to extreme thermal stress. Another problem is that, with the FGU design, the flange in the input side must be dismounted every time the char is taken out for analyses or for weight measurement. Therefore it was decided that the inlet and outlet side of the reactor must be redesigned. The major new design change for the SGU was to use machineable ceramic materials for both flanges and connectors.

An initial test was made of the SGU pyrolyzer with the new ceramic flanges installed. The results of a single SGU run are given in Table 2 for run #33. The hydrogen yield was relatively high compared to previous runs. Another improvement was that the product gases were "cleaner" with this reactor in terms of particulates, since no material was captured in the filter. The evolution rates for individual gases are presented in Figure 4 for this particular experiment.

Table 3 – Results from NASA Two-Stage Pyrolyzer Experiments with Chicken Manure Samples (wt.%, as-received basis)

Post Pyrolyzer Temperature	1100 °C	1100 °C	1100 °C	1100 °C	1100 °C	1050 °C
Run #	22	23	24	25	26	27
Char	41.1	40.7	40.8	40.8	40.7	41.1
H_2O	12.3	14.6	13.1	15.3	15.2	12.4
Carbon	0.8	2.6	9.2	2.8	4.1	9.1
Trap & Filter	4.7	4.5	2.9	2.8	3.9	14.1
Total Gases	41.0	38.2	37.8	38.6	36.3	23.4
H_2	1.6	0.6	0.5	1.2	0.7	1.1
C_2H_4	0.4	0.3	0.4	0.3	0.3	0.3
CH_4	3.0	3.0	3.3	3.6	3.0	1.7
CO_2	23.0	21.0	20.2	19.7	19.0	15.1
CO	13.0	13.3	13.4	13.8	13.3	5.2
NH_3	1.5	1.3	<0.1	1.2	2.4	1.2

Figure 5. Photograph of new sample holder and charred (unground) wheat straw sample.

ADDITIONAL REACTOR IMPROVEMENTS

When the raw waste (usually wheat straw) was fed into the FGU prototype in finely ground form, the resulting char was hard to clean from the reactor. However, if the feed was pelletized prior to the pyrolysis step, the resulting pieces of char were easily removable. For the SGU, a new sample holder was constructed in order to: 1) eliminate or minimize the pelletization step; 2) make it easier to insert and remove waste samples; 3) eliminate hot spots in the primary pyrolysis zone. The removable sample holder flange together with the stainless tube containing the sample is shown in Figure 5. Since the sample is held in this separate compartment, it was no longer necessary to press the sample into pellets. Instead, the sample was compacted without preliminary grinding into several "bales" of approximately the same diameter as the holder tube. Figure 5 also shows the charred remnant of a wheat straw bale sample after pyrolysis to 600 °C. The volume of the charred sample was reduced by about 50%.

The machineable ceramic flanges that are used in the SGU also tend to reduce the problem of plugging at the

reactor outlet, as discussed above. However, some problems remained: 1) The cement seals around the heating elements started to leak after only a few thermal cycles; 2) It is difficult to replace the heating elements in case of a heater failure. Consequently, it was decided to switch to some type of compression seal for these elements.

The available surface area on the flanges is relatively small, and does not allow for enough space to mount the ¾ inch fittings directly onto the flange. In case of the earlier stainless steel flanges, this was solved by welding on tube extenders, which moved away the heater ends from the "crowded" surface of the flanges. The other modification of the SGU was the large removable sample holder, which forces the three heaters on the input flange still closer to each other.

Based on the above considerations, the SGU design was further modified. The earlier 8 inch OD/4 inch ID reactor was replaced with a 5 inch ID reactor to provide enough space for the compression fittings. This increased the outer radiating (cylinder) surface from 700 square inches to 1000 square inches, thus significantly increasing the heat loss. This, in turn, increases the heat input required to run the reactor at 1100 °C. Since the 7 amp load on the heating elements is close to the maximum allowable current, the number of heating elements needed to be increased, leading again to potential overcrowding.

The approach that was taken was to improve the reactor thermal insulation. The most effective high temperature insulation uses a vacuum to minimize radiative and convective heat loss. For this application, a double-walled quartz thermal insulation jacket (similar to a Dewar container) was used, where the volume between the two cylinders is evacuated to prevent convective heat loss, and the inner surface of the outer quartz tube was coated with a reflective layer. The reflective layer of choice is a gold coating. However, it was difficult to apply a gold layer to the quartz cylinder and a decision was subsequently made to use an outside layer of aluminum. Even though the reflectivity of aluminum is lower, this will also provide a safety shield for the glass cylinders and will be much cheaper and longer lasting than a gold coating.

The reactor size modification, including addition of Dewar type thermal insulation, introduced several other changes:

- The outer stainless steel cylinder was not needed any more. This resulted in a significant weight reduction in prototype weight.
- The flanges must be sealed directly to the reactor tube. This will further simplify the design, since there is no need for inner and outer seals as well as the spring loaded compensation for heat extension of the reactor tube.

- Several other less important modifications were made concerning the flanges and the actual structure supporting the whole reactor system.

According to the original plan, the whole length of the reactor was to be insulated with a Dewar cylinder. However, there seemed to be the possibility of significant mechanical strain along the edges as a result of the high thermal gradients. Since the high temperature zone (~1100 °C) occupies only about half of the reactor length, it was decided to shorten the Dewar in order to remove this possibility. Therefore, the thermal insulation zone was divided into two sections. The shortened Dewar was evacuated to 1 mTorr and permanently sealed. Therefore, there is no need to run a vacuum pump during pyrolyzer operations.

SUMMARY AND CONCLUSIONS

Pyrolysis processing is one of several options for solid waste resource recovery in space. It has the advantage of being relatively simple and adaptable to a wide variety of feedstocks and it can produce several usable products from typical waste streams. The objective of this study was to produce a prototype mixed solid waste pyrolyzer for spacecraft applications. A two-stage reactor system was developed which can process about 0.5 kg of waste per cycle. This pyrolyzer has been successfully run with pure samples (wheat straw, chicken manure) and various mixed waste streams. Several improvements have been made in the reactor design in order to reduce weight and heat loss and improve the ease of sample loading and removal. The latest version of the prototype pyrolyzer, which can be considered the Third Generation Unit, awaits further testing.

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