Torrefaction Processing of Spacecraft Solid Wastes

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New technology is needed to collect, stabilize, recover useful materials, and store human fecal waste and other spacecraft solid wastes for long duration space missions. The system should also require minimal crew interactions, low energy demands, and tolerate mixed or contaminated waste streams. The current study addressed the technical feasibility of a torrefaction (mild pyrolysis) processing system that could be used to sterilize feces and related cellulosic biomass wastes (food, paper, wipes, and clothing), while simultaneously recovering moisture and producing small amounts of other useful products (e.g., CO_2, CO, and CH_4). This work was done using bench scale torrefaction processing units and examined different modes of heating (conventional and microwave) in laboratory studies. A fecal simulant was tested over a range of process conditions (temperature, holding time and atmosphere), along with selected runs with a sludge derivative (Milorganite), cotton fabric, and wipes. The results demonstrated that microwave heating allowed for careful control of torrefaction conditions for the fecal simulant. The net result was complete recovery of moisture, some additional water production, a modest reduction of the dry solid mass, and small amounts of gas (CO_2, CO, and CH_4) and hydrocarbon liquid production. The amounts of solid vs. gas plus liquid products can be controlled by adjusting the torrefaction conditions, especially the final temperature and holding time. The solid char product from the fecal simulant was a dry, free flowing powder that did not support bacterial growth and was hydrophobic relative to the starting material. The proposed torrefaction approach has potential benefits to NASA in allowing for solid waste sterilization and stabilization, planetary protection, in-situ resource utilization (ISRU) and/or production of chemical feedstocks and carbon materials. In particular, the torrefaction char residue has several potential applications in space. These include production of activated carbon, a nutrient-rich substrate for plant growth, construction material, radiation shielding, storage of elemental carbon, hydrogen, or oxygen, and fuel gas (CH_4, CO, and H_2) production.

Nomenclature

DAQ = Data Acquisition Card
DOC = Dissolved Organic Carbon
EMC = Equilibrium Moisture Content
FC = Fixed Carbon
FTIR = Fourier Transform Infrared
HMC = Heat Melt Compactor
ISRU = In-Situ Resource Utilization
LB = Lysogeny Broth
LPM = Liters per Minute

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MFC = Mass Flow Controller
MFM = Mass Flow Meter
RH = Relative Humidity
SBIR = Small Business Innovation Research
TG = Thermogravimetic Analyzer
TOC = Total Organic Carbon
UWMS = Universal Waste Management System
VM = Volatile Matter
WMS = Waste Management System
μ = Linear Attenuation Coefficient
ρ = Density

I. Introduction

A. The Problem and Technical Approach

New technology is needed to collect, stabilize, recover useful materials, and store human fecal waste and other spacecraft solid wastes for long duration missions both for crew safety, comfort and resource requirements and planetary protection [1-4]. This approach should also require minimal crew interaction, low energy demands, and tolerate mixed or contaminated waste streams. The current paper addresses a torrefaction (mild pyrolysis) processing system that can be used to sterilize feces and related cellulosic biomass wastes (food, paper, wipes, and clothing) and produce a stable char residue that can be easily stored or recycled, while simultaneously recovering all of the moisture and producing small amounts of other gases. The objective of the current study was to demonstrate the feasibility of this approach using bench scale experiments. This was accomplished in three tasks: 1) design and construct bench scale processing unit that can accommodate different modes of heating (conventional and microwave), 2) laboratory studies on a fecal simulant and related cellulosic biomass materials over a range of process conditions (temperature, holding time, and atmosphere), 3) evaluation of laboratory results and preliminary design of prototype system.

B. Potential Advantages versus Current Technology

The overall concept of a pyrolysis-centric Waste Management System (WMS), recently under investigation at Advanced Fuel Research, Inc. (AFR), is illustrated in Figure 1. Under previous projects, a pyrolysis-centric processing approach was proposed that could be implemented across a range of mission scenarios. For example, it can provide the same basic functions as the current Waste Management System (WMS), i.e. volume reduction, stabilization, and water recovery. In addition, it is also able to extract additional amounts of water and oxygen from most mixed solid wastes, which contain large amounts of oxygen. This approach can also be utilized to extract hydrogen and hydrocarbons from mixed solid wastes, which can be used in longer-term mission scenarios as fuel or for in-situ resource utilization (ISRU). Finally, it can produce carbon, which has many uses in long-term mission scenarios (e.g., flue gas clean-up, air revitalization, water purification, methane activation, hydrogen storage, radiation protection, ultracapacitors, etc.). AFR has developed the solid waste pyrolysis aspects of the above process under separate NASA-funded SBIR projects [5-8] and also a related SBIR project which focused on the utilization of the pyrolysis char to produce activated carbons for trace-contaminant removal [9]. The focus of the current project was to examine the feasibility of using mild pyrolysis (torrefaction) to maximize water recovery and minimize the formation of non-condensable gases and hydrocarbon liquids, while sterilizing and stabilizing fecal wastes and related cellulosic biomass wastes.

Another advantage of the pyrolysis-centric processing scheme shown in Figure 1 is that the individual stages of the process can be separated in time by minutes, days, or weeks, depending on the demand for the products that are being recovered from the waste. For example, adjusting the pyrolysis conditions to mild heating (torrefaction) conditions ($<300°C$) will significantly reduce the waste storage volume without significantly increasing the volume of gases that must be used, stored, or discarded. This is the objective of the torrefaction processing conditions that were studied under the current project. Under this study, torrefaction processing was applied to human fecal waste simulants and related solid waste streams. Similarly to pyrolysis processing, torrefaction processing can also provide the same basic functions as the current Waste Management Systems (WMS), i.e., volume reduction, stabilization, and water recovery. The torrefaction conditions can be adjusted (by increasing the final temperature and holding time) to extract hydrogen and fuel gases (CH_4, CO, H_2) from mixed solid wastes, which can be used in longer term mission scenarios. It can also produce activated carbon, which has many uses in, as noted above and described in more detail in Figure 2 for the torrefaction char residue.

WASTE-MANAGEMENT SYSTEM (WMS)
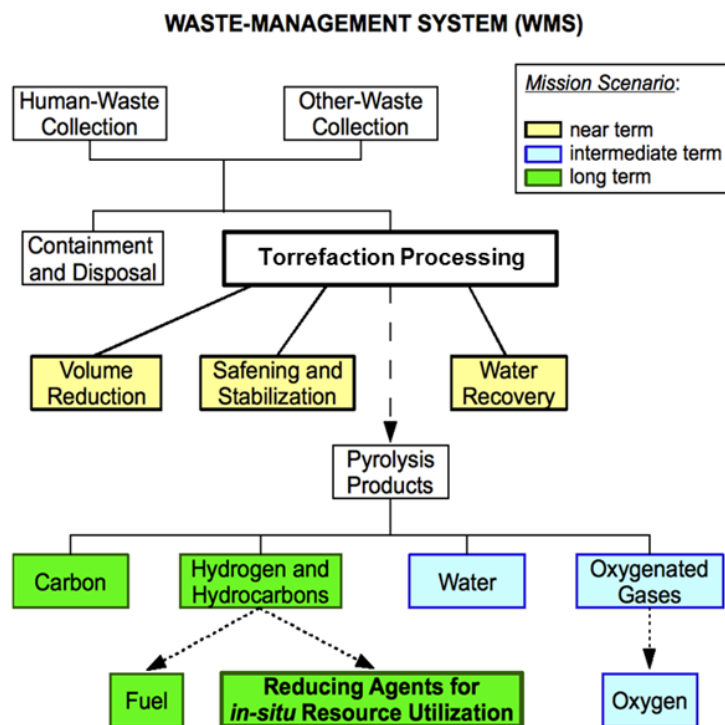
Figure 1. Schematic of Waste Management System (WMS) indicating how a pyrolysis/torrefaction-centric approach impacts near term, intermediate term and long term mission scenarios, including in-situ resource utilization.

C. Anticipated Benefits for NASA

The proposed methodology would make it technically feasible to process human fecal waste and related solid waste streams in space, which will benefit long term space travel such as an extended Lunar stay or a mission to Mars. As discussed above and indicated in Figures 1 and 2, the proposed torrefaction (mild pyrolysis) approach is beneficial to NASA in allowing for volume reduction, solid waste sterilization and stabilization, and water recovery for near term missions, while more severe pyrolysis processing would allow for enhanced water and CO_2 production, production of fuel gases (CH_4, CO, and H_2) and multi-purpose carbon, along with ISRU for longer term missions. The torrefaction processing system is also complementary to the Heat Melt Compactor (HMC) [10] and could also be designed to be compatible with the Universal Waste Management System (UWMS) [11], both now under development by NASA.

The primary purpose of future work will be to further evaluate torrefaction processing for NASA in the hope it that can ultimately be an important component of a Waste Management System (WMS). However, there are also important potential ancillary benefits in terms of increasing the technology base for stabilization and recycling of human fecal waste, as well as for biochar production from solid waste streams. The recent Gates Foundation "Reinventing the Toilet Challenge" supporting documents indicated that more than 2.5 billion people worldwide do not have access to safe and affordable sanitation [12]. The production of biochar from readily available cellulosic biomass and other waste materials has the potential to be important in efforts to promote soil fertility and carbon sequestration [13,14]. The support of such an effort is entirely consistent with NASA's objective of improving life on earth and being an incubator of dual-use technologies.
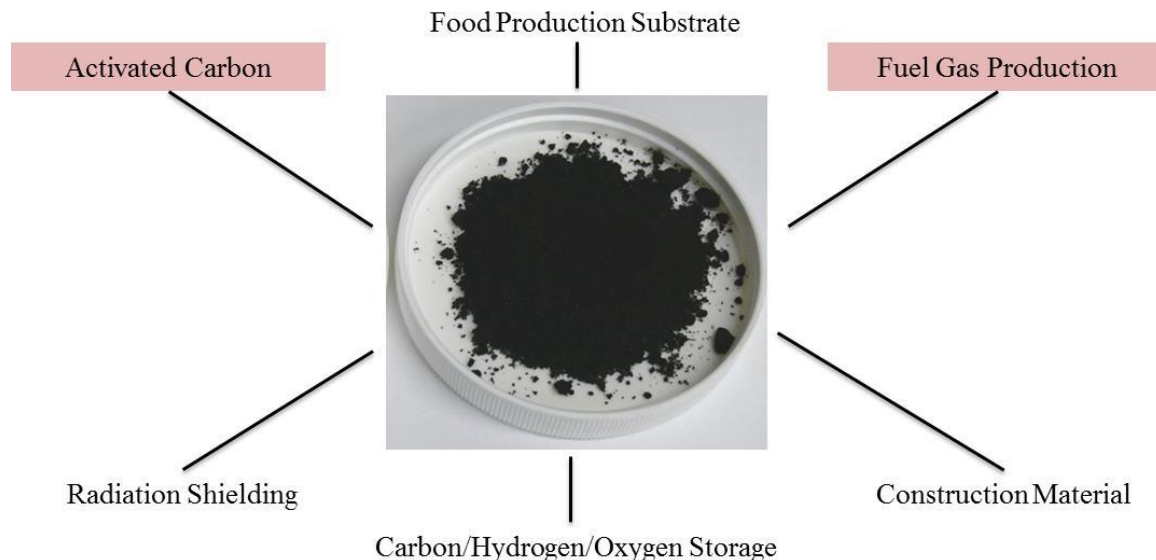
Figure 2. Spacecraft uses for torrefaction char residue. Shading indicates products that can be produced with additional heat treatment.

II. Background

Torrefaction can be viewed as pyrolysis performed under mild conditions [15]. It is usually defined as a thermochemical pre-treatment of biomass at 200–300 °C in the absence of oxygen. The typical heating rates used are lower than 50 °C/min, and the typical torrefaction time scales are one hour or less. Torrefaction has attracted a lot of interest in recent years, primarily in Europe, and the main reason seems to be a trend towards more sustainable power generation, in particular co-firing of biomass pellets with coal, but also rising fuel prices. A good overview of the torrefaction process and technologies can be found in Energy Research Center of the Netherlands (ECN) reports [16–18] that are available from the ECN web site. Torrefaction research is also carried out at the National Renewable Energy Center in Colorado (Nimlos *et al.* [19]), at the University of Leeds in the UK (Bridgeman *et al.* [20]), and also at the Royal Institute of Technology (KTH) in Sweden (Pach *et al.* [21]). The main advantages of torrefied biomass are: (1) higher energy density, (2) more homogeneous composition, (3) hydrophobic behavior, (4) improved grindability, and (5) elimination of biological activity.

III. Experimental

A. Experimental Apparatus

The initial work was to design and assemble a laboratory system for torrefaction studies on fecal simulant samples using different heating methods. A schematic of the torrefaction reactor system that was assembled for this project is shown in Figure 3. The primary components are the reactor unit (described below) and the gas analyzers, which include an On-Line Technologies, Inc. Model 2100 Process FTIR spectrometer and a Stanford Research Systems QMS 100 mass spectrometer. A two-stage condenser (25°C, 5°C) is employed for collecting condensable products (water and oil) and a wool filter is used to prevent residual particulate species from contaminating the sampling components of the gas analysis instrumentation. The FTIR gas analyzer has a frequency range of 700 – 6500 cm^{-1} with a resolution of 0.5 cm^{-1}. The instrument is calibrated for quantitative measurements of CO_2, CO, CH_4, C_2H_4 and H_2O (which is not completely condensed). The heated (125°C), single pass gas sampling cell (~ 50 cc volume, pathlength = 4 inches) has an exchange rate of 4-5 seconds and is ideal for observing transients in the various gas evolutions during the torrefaction experiments. The mass spectrometer is calibrated for H_2, CO_2, CH_4 and C_2H_2. For most of the experiments, the FTIR data were continuously collected at 10 second intervals and the mass spectra were collected every 13 seconds.
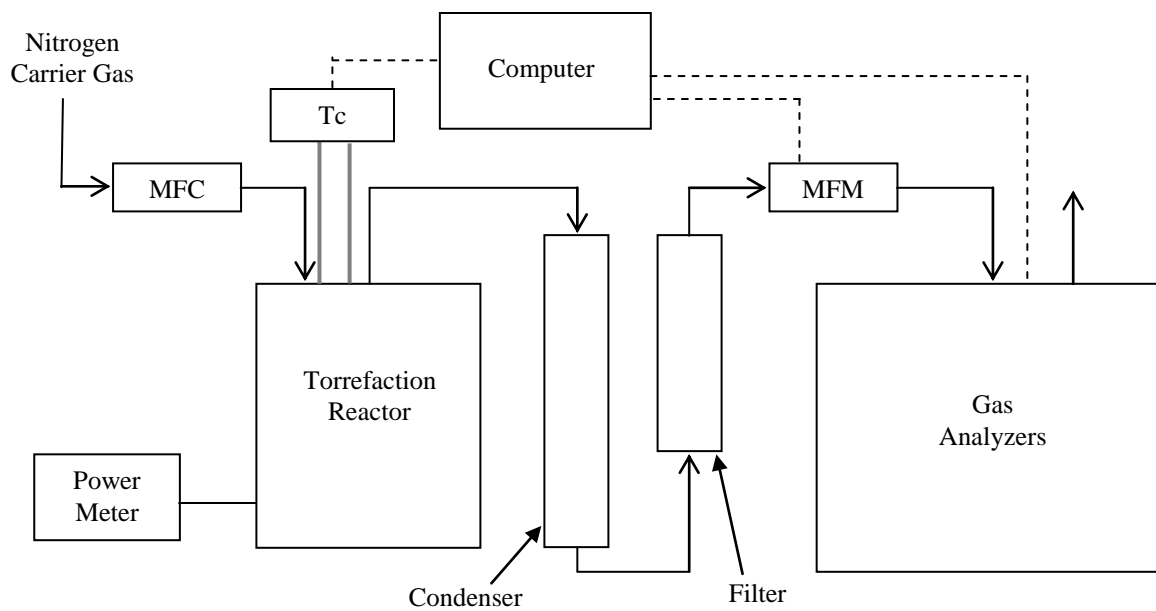
Figure 3. Schematic of the experimental apparatus employed for torrefaction experiments.

The nitrogen carrier gas is controlled by a mass flow controller (MFC) and a mass flow meter (MFM) measures the total gas volume exiting the reactor, as shown in Figure 3. The volumetric flow is recorded continuously throughout the experiment, along with the gas composition measurements. Since the MFM is calibrated for nitrogen, a correction factor (conversion factor) is applied to the MFM data based on the volume fraction of the measured gases, the manufacturer supplied conversion factors for the individual gases being quantified, and the total flow of nitrogen, which is held constant during each experiment. The flow rates for each gas are calculated based on the corrected total flow measured at the MFM and the gas concentration. Other features of the system include thermocouples for tracking the center and edge temperatures of the sample being torrefied and a power meter for recording the total electrical energy used during each torrefaction run.

A primary goal of this study was to evaluate two different heating approaches for torrefaction of the fecal simulant samples: 1) electrical heating using a conventional tube furnace-type heater and 2) microwave heating. The design and construction of both systems was completed and testing of each type of reactor unit was done. Figure 4 includes a schematic of the furnace reactor (top, left). As shown, the furnace is a vertically mounted ceramic radiant cylindrical heater (Watlow, 650 W) with a depth of 6 inches and an i.d. of 3 inches. The reaction vessel inserts into the furnace from the top and includes gas entry and exit ports. It also includes access for two thermocouples (0.125 inch dia.) for monitoring the sample temperature. One thermocouple monitors the temperature at the sample center and the "edge" thermocouple monitors the temperature at a radial distance of 0.75 inches (from center). Both thermocouples are inserted about halfway into the sample. For the earliest experiments, a quartz closed end tube (61 mm o.d. x 57 mm i.d.) sealed with a rubber stopper, was employed as the reaction vessel. Eventually, a stainless steel reaction vessel (60 mm o.d. x 57 mm i.d. x 125 mm insertion length) was fabricated. The metal vessel, shown in Figure 4 (top, right photograph) uses an O-ring face seal to provide a more reliable seal during heating. A photograph of the metal vessel seated inside the furnace is also shown in Figure 4 (bottom, left).

For microwave torrefaction experiments, a slightly modified microwave pyrolysis system was utilized that was developed for NASA in a recently completed SBIR Phase II program (Contract No. NNX10CA22C) [22]. This system was also recently employed for studying microwave pyrolysis of high fidelity waste simulants (HFWS) as part of a NASA-sponsored SBIR Phase III program [23]. The primary component of this system was a custom-modified industrial microwave oven from Microwave Research & Applications, Inc. [24]. The oven (model BP-211) provides 3.2 kW of microwave power (net output) using four air-cooled 800 W magnetrons (2450 MHz) and also incorporates four mode stirrers for enhanced field uniformity in the oven cavity. Although this system is greatly oversized (in power) for the sample sizes that were used (described below), it was chosen for this project because of its excellent heating uniformity and flexibility (power control). The oven was also designed to safely enable sample insertion into the cavity and extraction of product gases.

Tc (2)

Gas Products

Nitrogen
Carrier Gas

Reaction
Vessel

Tc

Radiant Ceramic Heater
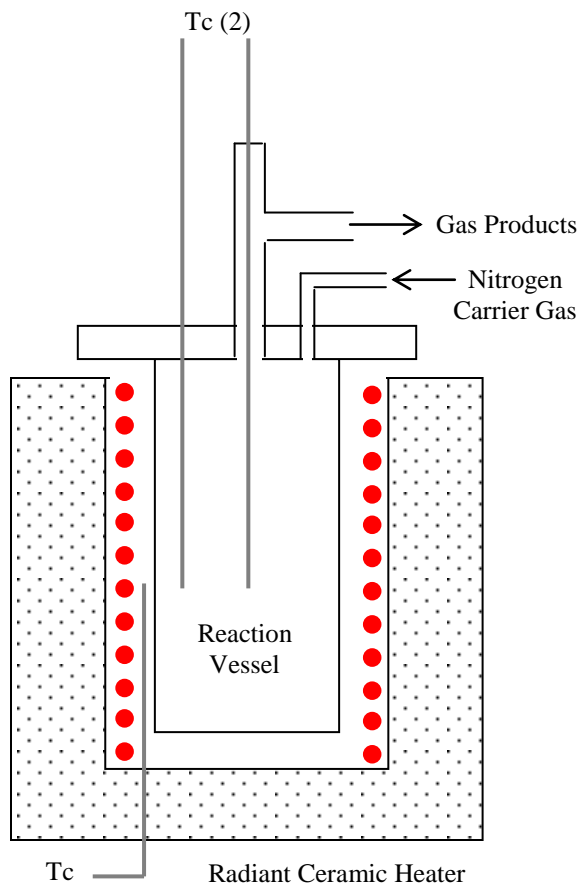
Figure 4. Top left: diagram of the furnace-heated torrefaction reactor. Top right: photograph of the stainless steel reaction vessel. Bottom left: photograph of the furnace-heated reactor and stainless steel reaction vessel.

The top flange is stainless steel since it is exposed to potentially corrosive gases during pyrolysis. The top flange also supports a thermoprobe that is inserted for temperature monitoring and control via a programmable temperature controller. The thermoprobe is basically a shielded thermocouple with a metal sheath that is grounded to the oven cavity.

For the current project, the torrefaction vessel used in the microwave oven was configured similarly to the geometry that was used in the furnace approach described above (Figure 4). The original quartz reaction vessel [8,22,23] was replaced with a closed end quartz tube (61 mm o.d. x 57 mm i.d. x 250 mm length) that is designed to mate with the top piston seal flange on the microwave reactor. The closed end tube was a commercial off-the-shelf component that was modified by a local glassblower to assure a gas-tight seal with the flange. Figure 5 displays a photograph of the microwave oven cavity configured with the torrefaction vessel. In keeping with the inside-out heating approach described previously [7,8], a SiC susceptor tube is located at the center of the quartz vessel. A thermocouple is shown inserted into the susceptor that was used for controlling the sample temperature. A new top flange was also fabricated for these experiments. It has separate gas entry and exit connections, as well as a center and edge thermocouple port, using the same geometry as the furnace reactor (0.75 inch spacing). An alumina tube provides additional support for the reaction vessel. Alumina, like quartz is relatively transparent (low-loss) to the microwave energy and therefore will not heat appreciably and add load to the system.

B. Materials

The composition of the base fecal simulant samples used for these experiments is shown in Table 1. This is similar to a formulation developed by Wignarajah et al. [25], the only difference being that *E.coli* addition was not used, for safety reasons. The water content, determined by drying in an 80°C oven for a period of 24 hours, is generally in the range of 20 – 23%, depending on the type of miso used. Because our "standard" miso source was temporarily unavailable, our initial three experiments used a low-salt alternative, which resulted in a simulant with slightly higher water content (~ 23%) than that obtained with the standard miso (~ 20%). Water was added to the "as prepared" simulant to generate levels up to 50% water content for torrefaction experiments.

Table 1. Chemical composition of fecal simulant.

Component	Weight Percent
Cellulose	14.3
Polyethylene Glycol	7.1
Peanut Oil	28.6
Miso	42.9
Potassium Chloride	5.7
Calcium Chloride	1.4

IV. Results and Discussion

A. Furnace and Microwave Heating Experiments

Under the current project, a total of 27 torrefaction experiments were performed, using both electrical furnace heating and microwave heating. The project focused mainly on fecal simulants, as described in the previous section, but other materials were also studied, including Milorganite (a sewage sludge-derived fertilizer product), baby wipes, and cotton fabric (from tee-shirts), using the microwave reactor. Furnace heating experiments involved approximately 125 – 170 g sample sizes, depending on the sample moisture content, while microwave heating experiments generally involved 250 – 335 g samples, again depending on the sample moisture content. In both cases, the sample was purged with a 1 LPM nitrogen carrier gas for a minimum of 30 minutes prior to heating and the carrier flow maintained throughout the course of the run. As described above, FTIR and mass spectra were collected and analyzed continuously throughout each run in order to track CO_2, CO, CH_4, C_2H_4, C_2H_2 and H_2O. After cool down, the reaction vessel, condenser, filter, and gas transfer lines were weighed to determine the remaining torrefied sample mass and condensate produced during each run. Mass balances were in the range 96 – 98 % for all but the first few runs. It should be noted that ethanol was typically also observed in the spectral data, but was not quantified. Based on its evolution beginning at low temperatures (<100°C), the ethanol is thought to be a fermentation product present in the miso, which represents the largest fraction of material in the simulant (excluding water). In addition, the spectra displayed features consistent with the presence of carbonyl compounds, such as

acetone and acetic acid. Both of these compounds were quantified in the small-scale thermogravimetic-FTIR studies discussed later in this paper.
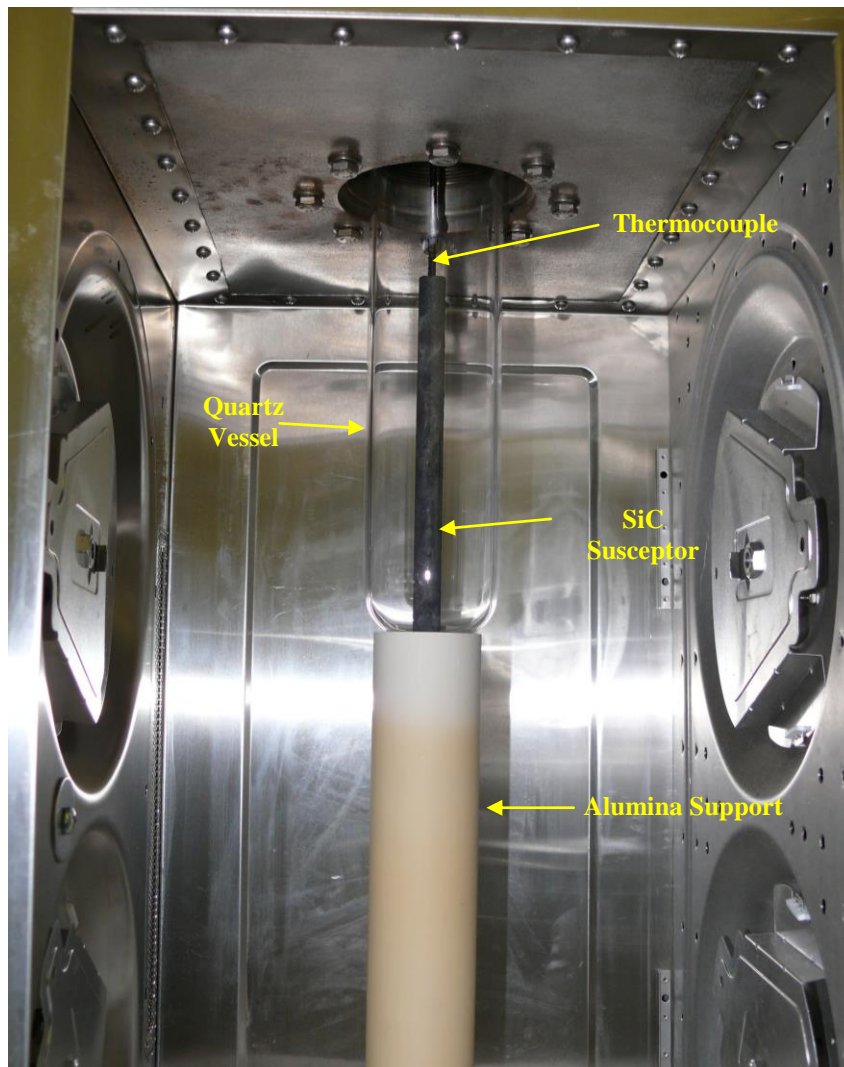
Figure 5. Photograph of the microwave system with the quartz torrefaction vessel.

As discussed above, two modes of heating were employed. In some cases, the furnace or microwave heater was temperature controlled, using feedback from one of the sample thermocouples or the furnace thermocouple, in the case of furnace heating. In other cases, the heater was operated in a constant power mode. For microwave heating, experiments were also conducted with and without the aid of a central microwave susceptor (SiC tube).

An example of a torrefied sample of fecal simulant is shown in Figure 6, which depicts a photograph of the untreated simulant (20% water content) along with the torrefied residue after heating to $\sim 300^{\circ}$C. The untreated sample has the consistency of moist sand. After heating, the simulant is fully charred and reduced to a mass of less than half of its initial (as-received) value. The amount of mass loss depends on the final temperature and holding time, as well as the initial moisture content.

Table 2 summarizes the results of the torrefaction experiments that were performed on the fecal simulant, Milorganite fertilizer, cotton fabric, and baby wipes. It includes the key experimental parameters, including the sample moisture content, electrical power, measured sample temperatures, and heating period. In addition, Table 2 lists whether or not a susceptor was employed in the cases where microwave heating was applied. The table also provides the solid, liquid, and gas yields, including the yields for CO_2, which was the major non-condensable gas observed in all runs. As described above, the solid and liquid yields are determined gravimetrically. The total gas yields are determined by difference, but these values may be susceptible to errors due to incomplete recovery of the

Table 2. Summary of gas, solid, and liquid yields for all torrefaction experiments. The rows are color-coded based on the type of material that was torrefied: no shading = fecal simulant, yellow = Milorganite fertilizer, blue = cotton material and pink = baby wipes.

Run No.	Sample Mass (g)	Moisture Content (%)	Heater Type	Heating Mode[1]	Susceptor	Elec. Power (W)	Max. Temp. (°C)	Ave. Temp.[2] (°C)	Heat Per.[3] (min)	Solid Yield (g)	Liquid Yield (g)	Gas Yield[4] (g)	Meas. CO$_2$ (g)	Elec. Energy (Wh/g)
1	126.3	23	Furn.	Temp.	-	375	300	I	I	81.9	34.1	I	2.55	1.61
2	125.1	23	Furn.	Temp.	-	240	300	I	I	77.2	35.6	I	2.95	1.60
3	125.2	23	Furn.	Temp.	-	240	521	>300	>30	38.1	71.3	I	5.08	1.60
4	125.6	20	Furn.	Temp.	-	240	310	272	61	65.2	51.6	8.77	3.45	1.75
5	125.2	20	Furn.	Temp.	-	240	310	264	71	52.9	59.9	12.42	4.53	1.76
6	250.2	20	Micro.	Temp.	SiC tube	Var.	342	I	I	162.9	77.6	9.65	5.05	1.46
7	250.2	20	Micro.	Power	SiC tube	1050	202	201	0.5	190.0	55.9	4.27	1.23	1.41
8	250.8	20	Micro.	Power	SiC tube	2100	274	220	15	185.0	60.4	5.40	1.84	0.88
9	250.5	20	Micro.	Power	SiC tube	2800	341	245	42	165.0	76.0	9.52	4.69	1.21
10	250.2	20	Micro.	Power	SiC tube	2218	304	232	35	173.0	68.4	8.82	3.20	1.19
11	276.9	20	Micro.	Power	none	2200	320	235	41	187.3	79.5	10.13	3.73	1.19
12	280.8	20	Micro.	Power	none	1680	304	237	41	193.3	77.2	10.23	4.07	1.18
13	335.9	40	Micro.	Power	none	1704	311	229	27	172.0	154.4	9.47	3.27	1.55
14	168.1	40	Furn.	Temp.	-	570	334	265	69	37.6	109.6	20.88	8.07	1.96
15	167.9	40	Furn.	Temp.	-	240	211	203	9.7	89.0	72.2	6.70	1.33	1.79
16	336.1	40	Micro.	Power	none	1264	235	< 200	L	187.8	141.0	7.27	1.49	1.66
17	168.7	40	Furn.	Power	-	345	303	228	38	48.2	106.4	14.12	4.76	1.69
18	327.9	50	Micro.	Power	none	1254	266	< 200	L	151.8	168.7	7.44	0.89	2.15
19	327.9	50	Micro.	Power	none	1258	303	232	26	145.1	173.1	9.62	2.01	2.15
20	267.2	40	Micro.	Power	none	1262	293	240	10.8	147.5	118.1	1.62	0.94	1.85
21	327.7	50	Micro.	Power	none	1256	284	232	53.4	144.7	172.0	10.98	2.04	2.57
22	64.2	4.5	Micro.	Power	SiC tube	1013	278	239	31.5	56.4	3.8	4.09	0.78	6.35
23	74.6	4.5	Micro.	Power	SiC tube	1263	314	279	97.3	42.5	21.4	10.79	3.40	10.52
24	163.7	81	Micro.	Power	SiC tube	1257	321	248	62.6	29.3	129.7	4.67	0.65	9.63
25	300.3	40	Micro.	Power	SiC tube	1263	341	268	80.1	153.2	141.6	5.48	4.17	2.94
26	301.6	40	Micro.	Power	SiC tube	1275	355	280	85.8	148.1	141.9	11.51	4.06	2.87
27	302.9	40	Micro.	Power	SiC tube	1260	317	272	76.2	150.5	142.9	9.54	3.55	2.83

1. *Temp. = Temperature Controlled, Power = Constant Power*
2. *Average sample temperature where both sample temperatures are ≥ 200 °C. I = Insufficient data.*
3. *Period during which both sample temperatures are ≥ 200 °C. I = Insufficient data. L is marked where one of the thermocouples did not reach 200 °C.*
4. *Determined by difference. I = Insufficient data.*

solids and/or liquids. Finally, the table provides the calculated electrical energy usage for each experiment, on a per gram sample (wet) basis.

Figure 7 displays the gas evolution profiles for CO, CO_2, CH_4 and C_2H_4 during furnace torrefaction of an ~ 125 g sample of simulant (top) and microwave torrefaction of an ~ 250 g sample of simulant (bottom), both at a nominal 300°C maximum sample temperature. Also shown are the sample center temperature and edge temperature time traces measured for each case. For the furnace run, where the setpoint temperature is 300°C, the "control" thermocouple is a thermocouple inserted about halfway into the sample, near the edge of the reaction vessel. For the microwave case, a constant power heating mode was employed, with the microwave power held at ~ 1700 W (electrical). Observations include:

- The sample begins heating much faster during microwave processing.
- Center and edge temperatures track much closer during microwave processing, indicative of more volumetric heating. In contrast, heat transfer to the center of the sample during furnace heating is dominated (limited) by conduction.
- The microwaved sample begins cooling right after shutdown (~13 minutes) while the furnace heated sample center temperature continues to rise over the next 12 minutes after shutdown (~ 80 minutes).
- CO_2 is the dominant non-condensable gas produced.
- In both cases, CO_2 evolution was first observed at temperatures around 100°C.
- CO appears at a lower temperature in the furnace case, but this may be because the highest temperatures are not being measured, which are closest to the reaction vessel wall.
- Methane and ethylene were only observed in the furnace case under these conditions (maximum temperature of ~ 300°C).

Figure 6. Photograph of a sample of undried fecal simulant (left) and a torrefied (~300 °C) sample (right).

As shown in Figure 8, which plots CO_2 production (top) and char fraction (bottom) as a function of the maximum measured sample temperature, more CO_2 is produced at higher temperatures while, conversely, less char remains after higher temperature processing. Regarding the heating method, microwave processing *appears* to generate less CO_2, although this observation may be due to some measurement bias in the temperature data, as discussed above. Note that one of the furnace data points (334°C) appears as an outlier, exhibiting a relatively high CO_2 yield in conjunction with a low char fraction. For this experiment, the furnace was run at a high temperature (at high electrical power), which may have resulted in a higher temperature gradient in the sample and/or some thermal runaway near the vessel walls.
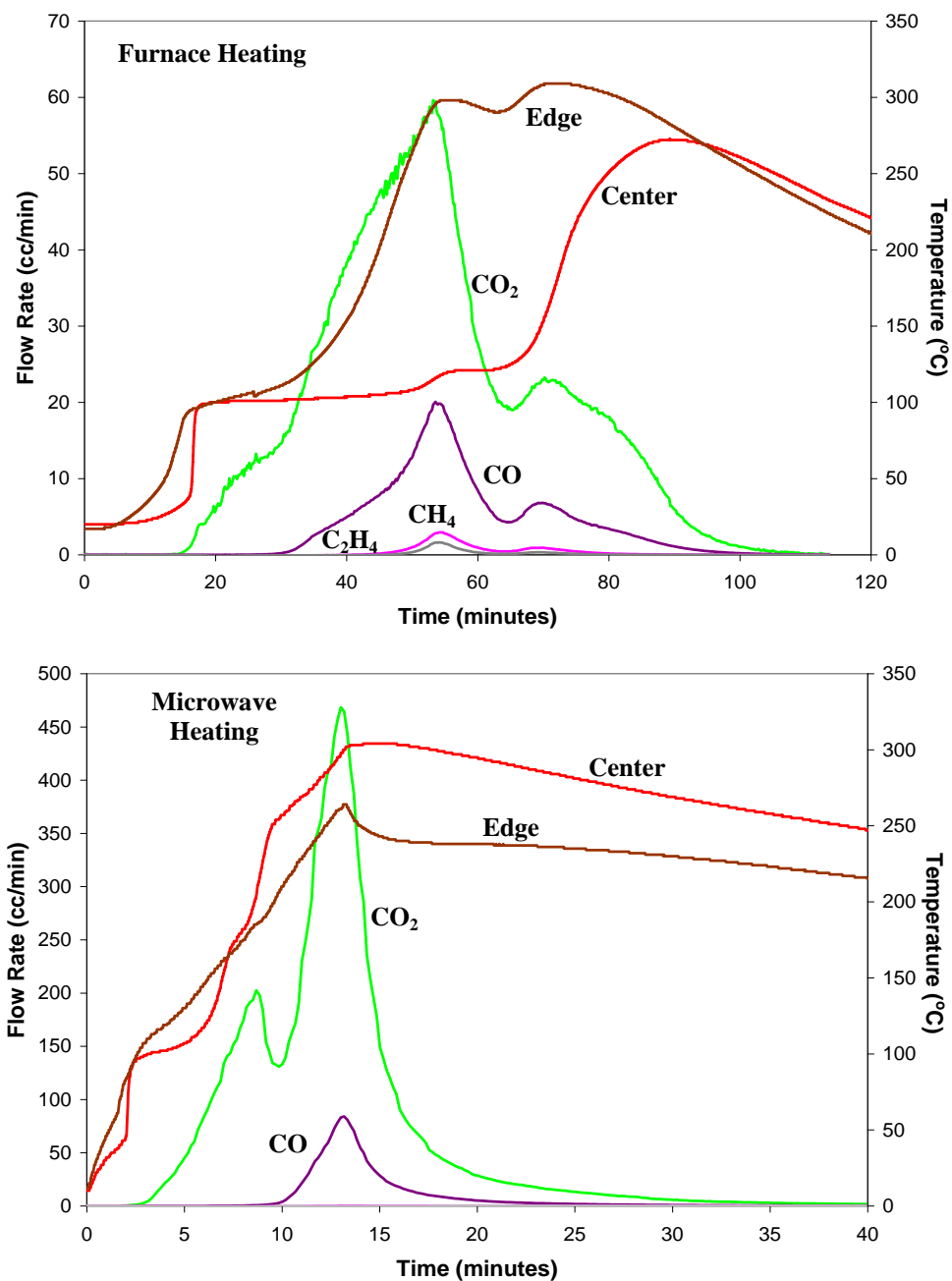
Figure 7. Gas evolution rates measured for CO_2 (green), CO (purple), CH_4 (pink) and C_2H_4 (gray), during torrefaction of the fecal simulant using furnace heating (top) and microwave heating (bottom). Also shown are the sample center temperature (red) and edge temperature (brown) time traces.
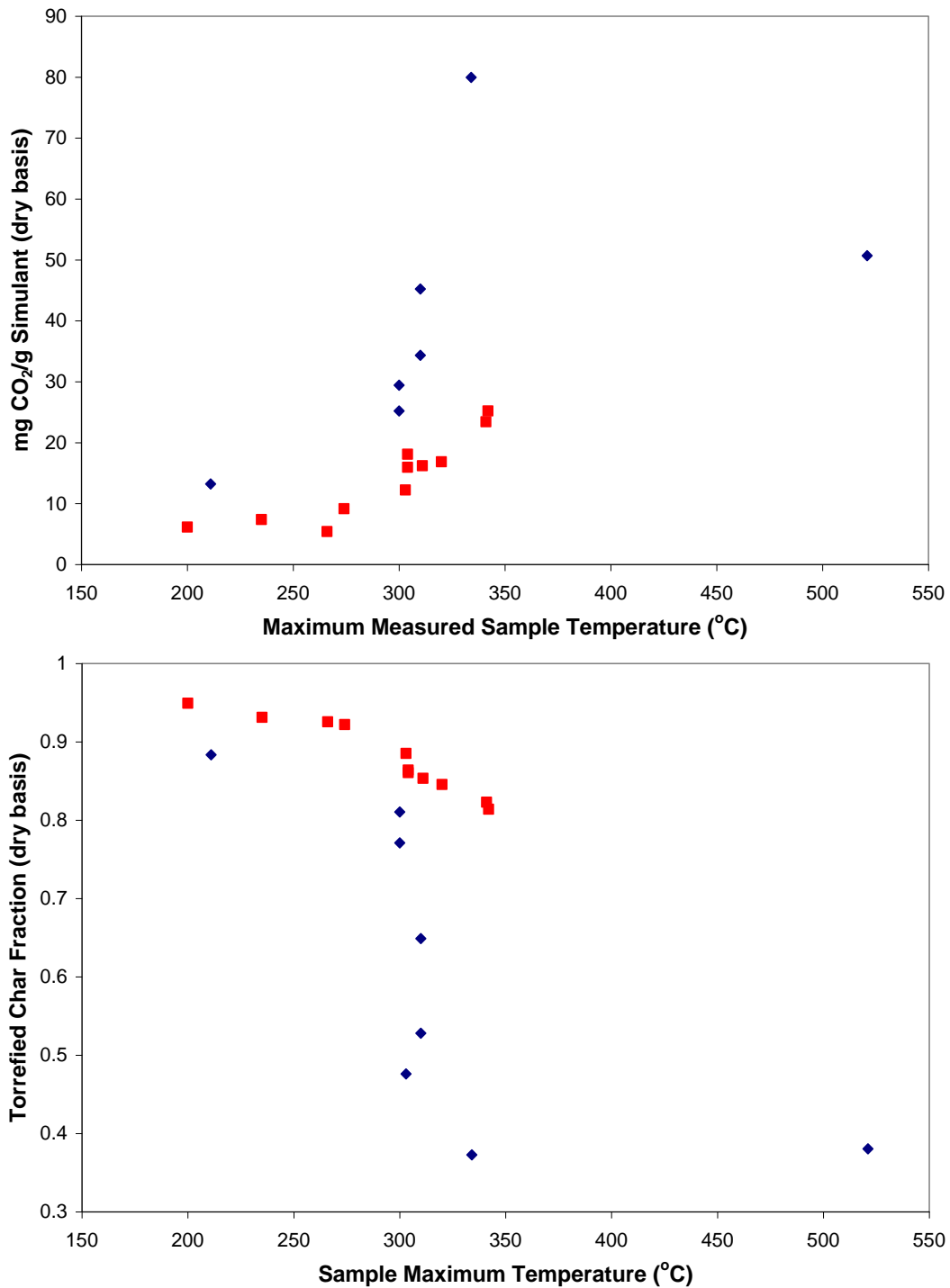
Figure 8. CO_2 production (top) and torrefied char fraction (bottom) as a function of the maximum simulant sample temperature during torrefaction. Microwave data are shown in red and furnace data are shown in blue.

The data in Table 2 indicate that the use of a susceptor material for microwave heating of the fecal simulant appears to offer no advantages in terms of the heating efficiency, compared to experiments where the susceptor was not employed. In addition, the energy efficiency of the furnace and microwave heating systems appear to be comparable for processing the fecal simulant, after adjusting for sample size differences. However, it is believed that an optimized microwave heating approach is a potentially more efficient method of heating, as discussed in more detail below.

Of the other materials that were studied, the Milorganite proved to be the easiest material to heat using microwave processing. However, after heating it to a temperature of 293°C, it appeared visually unchanged. It was subsequently learned that, during manufacturing, Milorganite is processed at much higher temperatures, ranging from ~480 to 650°C. On the other hand, the cotton material proved to be more difficult to heat and was not converted to a powder at the temperatures that were explored. For Run #22 (max. temp. = 278°C), the temperatures were not sufficient to completely convert the cotton material to char. For Run #23, the torrefaction temperature was increased to 314°C and maintained for a much longer period, resulting in nearly complete charring of the tee-shirt material and ~ 50% weight loss, although the fabric structure still remained largely intact. Baby wipes showed similar results to the cotton using microwave treatment. Both of these materials are mostly cellulose and do not have the hemi-cellulose content that initiates the early decomposition and nearly complete disintegration of materials that contain whole biomass, such as the fecal simulant or plant biomass materials, under torrefaction conditions [15].

Runs #25 and #26 were done under the same nominal conditions and show good agreement, except for the total gas yield, which is determined by difference and will be influenced by any mass balance closure issues. The final experiment that was performed (Run #27) examined the effect of the carrier gas composition during microwave heating of the fecal simulant by substituting air for nitrogen. When compared to an experiment in nitrogen under similar conditions of temperature and heating period (Run #25), the solid, liquid and CO_2 yields are quite similar. This result suggests that, at least for the fecal simulant, torrefaction is relatively insensitive to the carrier gas atmosphere (nitrogen vs. air).

B. Char and Liquid Analysis

Several samples of char and liquids derived from torrefaction of fecal simulants were analyzed by Huffman Laboratories, Inc. (Golden, CO), to assess their composition and organic content, respectively. Table 3 lists the results for the solids analyses, showing the elemental composition (C,H,N,O,S) as well as ash, Volatile Matter (VM) and Fixed Carbon (FC), along with temperature data measured for each run. It also lists the starting composition for a raw feces simulant used in a previous project, but made with the same recipe. Note that the data are listed in order of increasing maximum torrefaction temperature. In general, the compositions for the four samples generated at temperatures under 285°C are quite similar and only modestly different than the starting material. The fact that the char product from the torrefaction process retains a lot of its hydrogen content suggests that it might be better at radiation shielding than activated carbon, although not as good as polyethylene [26]. Some preliminary measurements were made in this regard and are discussed below (see Table 7). For the highest temperature case, higher nitrogen and ash and a decrease in carbon, hydrogen, oxygen, and VM are observed.

Table 4 lists the results for the liquid analyses in terms of Total Organic Carbon (TOC) and Dissolved Organic Carbon (DOC). For all samples, particulate carbon represents about 5% or less of the TOC. The table also shows the TOC values normalized to a moisture content of 50%, the highest moisture content that was studied in this project. As was the case for the solids analyses, the four samples generated at torrefaction temperatures less than 285°C are similar in terms of their normalized TOC, ranging from 0.62 – 0.77 %. Some of the organics may be condensed ethanol that is thought to originate from the miso in the simulant, as discussed above. Nonetheless, these values are very close to the acceptable limits for water recycling applications in space. It should be noted that the 50% moisture content used for the normalized TOC measurements is actually on the low end of the expected moisture contents in human feces (65-85 %) [27]. The modest TOC amounts confirm that most of the liquid product collected is water and that the amount of water is in excess of the moisture content (see Table 2). In fact, excluding the highest temperature runs, the amount of liquid collected in excess of the nominal moisture content for the fecal simulant ranged from 10-100%, with an average of ~20%.

Table 3. Composition of char from several fecal simulant torrefaction experiments.

Run #	Moisture Content (%)	Max. Temp. (°C)	Ave. Temp.[1] (°C)	Composition (%)							
				C	H	N	O	S	Ash	VM	FC
RAW	19	—	—	49.70	7.77	0.97	28.15	0.06	13.35	75.60	11.05
7	20	202	201	52.14	7.46	1.04	25.03	0.08	14.25	73.69	12.06
15	40	211	203	56.68	7.65	1.05	19.63	0.06	14.93	70.61	14.46
16	40	235	L	53.33	7.55	1.02	23.02	0.06	15.02	73.15	11.83
21	50	284	232	55.83	7.61	1.09	19.39	0.06	16.02	71.39	12.59
14	40	334	265	47.33	4.66	1.59	11.76	0.05	34.61	37.11	28.28

1. *Average sample temperature where both sample temperatures are ≥ 200 °C. L is marked where one of the thermocouples did not reach 200°C. The analyses are reported on a dry basis, except for moisture (as received).*

Table 4. Total organic carbon and dissolved organic carbon of liquid yield from several simulant torrefaction experiments.

Run #	Moisture Content (%)	Max. Temp. (°C)	Ave. Temp.[1] (°C)	Total Org. Carbon (mg/L)	Dissolved Org. Carbon (mg/L)	Normalized[2] Total Org. Carbon (%)
7	20	202	201	17000	16400	0.68
15	40	211	203	9630	9010	0.77
16	40	235	L	7700	7680	0.62
21	50	284	232	7490	7170	0.75
14	40	334	265	29000	28000	2.32

1. *Average sample temperature where both sample temperatures are ≥ 200 °C. L is marked where one of the thermocouples did not reach 200°C.*
2. *Normalized to 50% moisture content.*

The results of these liquid analyses are consistent with visual observations during torrefaction processing. In general, heavy oils and tars are not observed in the reactor condensers until temperatures approach ~ 300°C. As indicated in Figure 3, there is an initial condenser, which consists of an air-cooled glass tube loosely filled with coarse, stainless steel wool. At 70°C, water is observed collecting in the condenser, along with a relatively small amount of reddish fluid that has not been identified. Evolution of the reddish condensate is fairly brief, ending when the sample temperature reaches about 100°C. As the reaction progresses up to about 290°C, the condensate produced is fairly clear and colorless, causing the color of the liquid in the condenser to gradually fade. As the sample temperature continues to increase beyond 290°C, tar and/or oil becomes noticeable.

C. Simulation of Torrefaction Process

The use of TG-FTIR analysis (programmed pyrolysis in a TGA with FTIR analysis of evolved gases) was applied to the fecal simulant at 30 K/min to 900°C and the cumulative amounts of product yields at an intermediate temperature of 300°C were compared to the final product yields at 900°C. The data can be found in Table 5 and are largely consistent with the torrefaction results. In particular, it confirms that about half the pyrolytic water can be produced under torrefaction conditions, presumably due to dehydration of hemi-cellulose.

D. Biological Activity Testing

Some of the advantages of biomass torrefaction include elimination of biological activity and increased hydrophobic behavior. In this case, methodologies were established for evaluating these characteristics of the torrefied fecal simulant samples that were produced in this project. In order to assess the biological activity, agar plates were dusted with a small quantity of the torrefied powder and monitored for evidence of biological growth, such as mold. The agar plates were commercial lysogeny broth (LB) agar plates manufactured by EZ BioResearch LLC. After 15 days, abundant mold colonies were observed in the untreated simulant sample, while none were observed in the torrefied samples.

Table 5. Results for TG-FTIR analysis of fecal simulant at 300°C and 900°C while heated at 30K/min.

Sample:	Standard Feces Simulant			
Heating Rate:	30 K/min (300 °C)		30 K/min (900 °C)	
Run No.	AFR5722		AFR5772	
TG-FTIR Profile Used:	BIOM30		BIOM30	
Drying Régime:	30 min at 80 °C		30 min at 80 °C	
Sample Size (mg):	54.81		54.81	
	Product Yield (wt%)			
	at 300 °C (a.r.)	at 300 °C (daf)	at 900 °C (a.r.)	at 900 °C (daf)
Moisture	17.53		17.53	
Ash (a.r.)	0		0.00	
Ash (dry)	0.00		0.00	
VM (from TGA data)	10	12.13	65.81	79.80
Char	72.47	87.87	16.66	20.20
Tar (by difference)	3.35	4.06	46.70	56.63
Water (pyrolytic)	4.30	5.22	9.28	11.25
Carbon Monoxide	0.07	0.08	1.42	1.72
Carbon Dioxide	0.90	1.09	3.47	4.21
Methane	0.02	0.02	0.73	0.88
Ethylene	0.00	0.00	0.09	0.11
Hydrogen Cyanide	0.01	0.01	0.15	0.18
Ammonia	0.00	0.00	0.09	0.11
Isocyanic Acid	0.00	0.00	0.06	0.08
Formaldehyde	0.01	0.01	0.51	0.62
Acetaldehyde	0.00	0.00	1.29	1.56
Formic Acid	0.06	0.07	0.31	0.37
Acetic Acid	0.33	0.40	0.27	0.33
Methanol	0.00	0.00	0.00	0.00
Sulfur Dioxide	0.00	0.00	0.00	0.00
Carbonyl Sulfide	0.01	0.02	0.07	0.08
Phenol	0.00	0.00	0.00	0.00
Acetone	0.12	0.15	1.40	1.70

Table 6. Equilibrium moisture content results for dried simulant samples and several char samples. The temperatures provided with each char sample are the average temperature above 200°C and the time period is the period during which the sample temperature was higher than 200°C.

Sample	Equilibrium Moisture Content (%)			
	RH = 11.3%	RH = 43.1%	RH = 57%	RH = 73.1%
Simulant, Desiccator-dried	1.97	4.33	-	-
Simulant, Oven-dried (80°C, 24 hr)	0.84	3.53	6.49	32.52*
Char, Run # 7, (201°C, 0.5 min)	-	2.07	3.28	-
Char, Run # 15, (203°C, 9.7 min)	0.43	1.61	-	24.94*
Char, Run # 8, (220°C, 15 min)	0.53	1.58	3.16	28.72*
Char, Run # 9, (245°C, 43 min)	-	1.48	2.13	22.93*
Char, Run # 3, (>300°C, >30 min)	-	2.40	3.18	-

Moisture content after four weeks (equilibrium not reached).

Table 7. Mass attenuation coefficients (μ/ρ) measured for various materials including torrefied fecal simulant.

Sample	Measured μ/ρ (cm²/g)	Published* μ/ρ (cm²/g), ~ 700 keV
Torr16	0.057	-
Torr21	0.055	-
Simulant (unheated)	0.048	-
Water	0.054	0.084
Graphite	0.046	0.076
Polyethylene	0.057	0.086

* Reference 28.

E. Hydrophobicity (EMC) Testing

Hydrophobicity testing of various torrefied fecal simulant samples was also done. The hydrophobicity was evaluated by determining the Equilibrium Moisture Content (EMC) of torrefied samples at various relative humidity levels, using procedures described in reference 29 (and references therein). The methodology involves placing a dry sample of material in a glass petri dish and then placing the dish in a sealed mason jar containing a saturated aqueous salt solution. Different salts are employed to vary the relative humidity (RH) levels in each jar including lithium chloride (RH = 11.3%), potassium carbonate (RH = 43.1%), sodium bromide (RH = 57%), and sodium chloride (RH = 73.1%). The sample dish is placed on top of a plastic standoff to keep it separated from the solution. The jars are maintained in a temperature-controlled enclosure (28 to 30°C) for a period of up to 4 weeks and the weights of the samples are periodically checked until they are stable. After the sample weight has stabilized, or by the end of four weeks, the sample is oven dried at 80°C for 24 hours to determine the moisture content.

Table 6 compares EMC results for several samples of the char residue after different torrefaction conditions. The table also includes results for samples of the as-received simulant that were dried in a nitrogen-purged desiccator, as well as samples oven-dried in air at 80°C. Overall, it can be seen that the torrefied samples are more hydrophobic than the dried feces simulant samples, with the most hydrophobic samples being from Run #9. The hydrophobicity increases with torrefaction temperature, except in the case of Run #3, where temperatures as high as 521°C were measured (Table 2). In this case, where the reaction crossed over into the pyrolysis regime, the char is less hydrophobic. Note that the values shown for RH = 73.1% are not the true EMC of these samples, since their weights had not yet stabilized after 4 weeks. Nonetheless, it can be seen that the moisture uptake at this highest level is slower for the torrefied samples.

F. Radiation Shielding

Some preliminary attenuation measurements were conducted to assess the feasibility of using torrefied feces for shielding of gamma rays. The measurements were performed by inserting polypropylene containers filled with different simulant samples between a Cesium-137 point radiation source (primary peak ~ 662 KeV) and a Black Cat Systems ionizing radiation detector. The linear attenuation coefficient, μ, was then calculated from the transmittance measurements (using an empty container as a reference) for each sample. Table 7 displays the mass attenuation coefficients, μ/ρ, (ρ = sample density) measured for two torrefied samples of fecal simulant, in which the biomass was heated to temperatures of 235°C (Torr16) and 284°C (Torr21). The table also includes measurements for an unheated fecal simulant sample with ~ 20% water content, as well as pure water, polyethylene and carbon (graphite), to provide a relative evaluation of the biomass shielding performance. For validation of these measurements, published values from NIST [28] for the three standard materials are provided. Relatively speaking, it can be seen that the measurements of μ/ρ for the three standard materials follow the same trend as the published data, with polyethylene being slightly higher than water which, in turn, is higher than graphite. There is, however, a significant discrepancy in their magnitudes, which may be due to differences in the measurement methodology, for example. More importantly, however, it can be seen that the torrefied biomass provides shielding performance comparable to the water and polyethylene, in this energy range. The data also suggest that there may be some improvement in shielding for the torrefied material as compared to the unheated simulant. While these measurements were performed at an extremely low end of the energy range for galactic cosmic radiation, they do suggest some potential for torrefied human feces as a radiation shielding material in deep space environments.

G. Evaluation of Laboratory Results and Preliminary Design of Prototype

The experiments performed, employing both microwave and electrical furnace heating, have demonstrated that torrefaction provides an effective means of processing a fecal simulant biomass material in terms of elimination of biological activity, significantly increased hydrophobicity of the resulting char material, minimal gas production, and enhanced water recovery with low organic contamination. These results also indicated that microwave heating provides faster, more volumetric heating (even heating), with superior control, when compared to furnace heating.

In terms of energy efficiency, both heating methods were shown to be fairly comparable. As an example, Run #1 (furnace heating), from Table 2, had a maximum temperature of 300°C and a solid yield of 65%, with an energy usage of 1.61 Wh/g for the fecal simulant with a moisture content of ~ 20%. For Run #10 (microwave heating), the maximum temperature was similar (304°C), with a slightly higher solid yield (69%), but with a slightly lower energy usage of 1.21 Wh/g, also for the fecal simulant with a moisture content of ~ 20%. Although the furnace system was not optimized and could be improved in terms of its energy efficiency, it is believed that the potential for significantly improving the energy efficiency using a microwave approach is much higher. As mentioned above, the microwave system that was employed for the current effort was oversized compared to the sample sizes that were being studied, in both size and power.

Table 8. Heating efficiency calculations based on water calorimetry measurements inside the microwave oven cavity that was employed for the microwave torrefaction experiments.

Water Volume (L)	Electrical Power (W)	Adsorbed Power (W)	Heating Efficiency
0.3	1000	315	0.32
0.9	820	315	0.38
2.4	917	415	0.45
2.4	5351	3084	0.58

In order to better understand the implications of heating these relatively small samples (~ 0.3 L) in the large microwave oven cavity (41.5 L), some simple calorimetry measurements were performed on different volumes of water at different microwave oven power settings. The temperature rise of a specific quantity of water was measured for a specific time period, while monitoring the electrical power of the oven. The overall "heating efficiency" was then calculated by the ratio of the power absorbed by the water sample and the measured electrical power. Table 8 summarizes the results from these measurements and two effects are quite obvious. First, it can be seen that the heating efficiency increases substantially with sample size, which shows evidence of a geometric effect. In addition, the heating efficiency also appears to improve at the highest power level, suggesting that the magnetrons' conversion efficiency may be somewhat power dependent. Running the system with a more appropriately matched load at maximum power results in a nearly two-fold increase in the heating efficiency and this level of improvement can be expected in an optimized microwave reactor for torrefaction of biomass.

V. Future Work

Based on the above results, a preliminary design for a prototype microwave-based fecal waste torrefaction system was developed that would be compatible with the Universal Waste Management System (UWMS) currently under development at United Technologies [11]. In that system, fecal waste is collected in a single bag, which is then collected in a metal canister (containing multiple bagged samples). In this preliminary design, the metal canister, shown in red, would be attached to the microwave torrefaction reactor (2450 MHz), as shown schematically in Figure 9. In this geometry, the canister conveniently becomes part of a microwave multimode oven cavity, which eliminates the need to transfer the bagged waste samples into a fragile, microwave-compatible vessel, such as quartz or ceramic. The reactor/canister joint would be designed to prevent both gas and microwave leakage, using appropriate gas seals/choke designs. Multimode oven cavities are usually rectangular boxes in shape, with at least one dimension several half-wavelengths in length. The UWMS canister is currently a cylindrical container, but it is expected that changing the canister design to have a square cross-section would not be an issue.

Note that the design calls for a sealed microwave-transparent window that prevents gases from contaminating the microwave generator. The window material would likely be Teflon or glass, both of which are exceptionally transparent to microwaves. Teflon has additional advantages in that it is non-breakable and has excellent non-stick properties. Additional options (not shown) would include the incorporation of a mode-stirrer device, to improve the field uniformity in the oven cavity and/or the use of some type of mechanical device to agitate the waste sample. If necessary, the UWMS waste canister could include a bottom liner made of silicon carbide, to assist in the heating as a microwave susceptor layer. However, based on the current results obtained with the fecal simulant, this would probably not be required. The final prototype would likely be equipped with gas analysis instrumentation for monitoring and/or controlling the torrefaction process.

VI. Conclusions

The current project demonstrated that microwave (volumetric) heating allowed for careful control of torrefaction conditions for a fecal simulant and related cellulosic biomass materials. The net result was complete recovery of moisture, some additional water production (~20% higher than the nominal moisture content), a modest reduction of the dry solid mass and small amounts of gas (CO_2, CO, CH_4) and hydrocarbon liquid production. The amounts of solid vs. gas plus hydrocarbon liquid products can be controlled by adjusting the torrefaction conditions (final temperature, holding time). The use of air as the purge gas gave similar results as nitrogen for the fecal simulant. The solid char product from the fecal simulant was a dry, free flowing powder that did not support bacterial growth and was hydrophobic relative to the starting material. At temperatures less than ~290°C, the condensate was fairly clear and colorless and had a moderate total organic carbon content (~0.7%). In addition, the torrefaction method can be applied to other types of wet or dry cellulosic biomass (food, paper, wipes, and clothing), which provides for
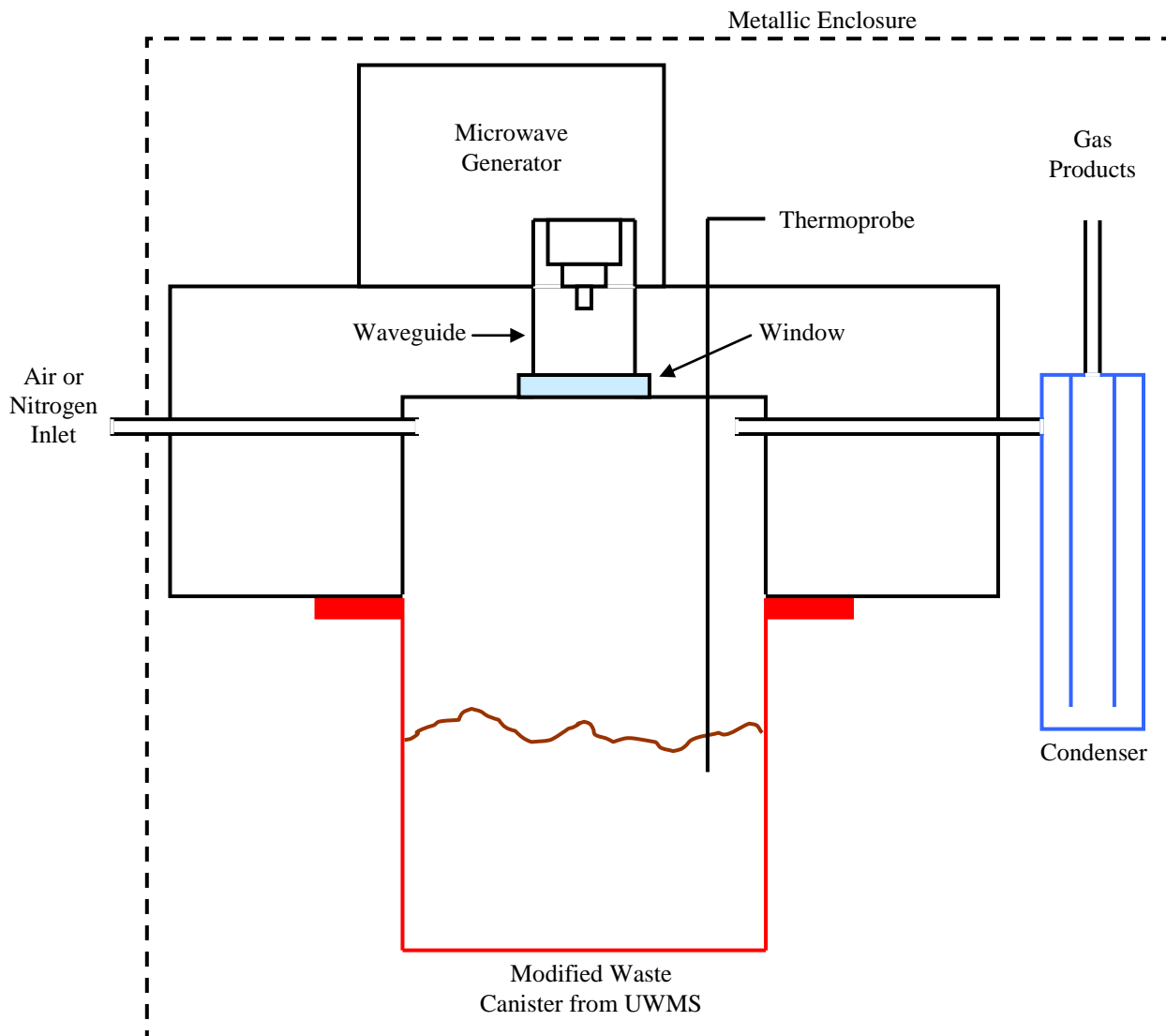
Figure 9. Schematic of a preliminary prototype microwave reactor for processing waste from the UWMS. The UWMS metal canister with waste is shown attached directly to the reactor in a manner such that the canister becomes part of the multimode reactor cavity.

some desirable redundancy in the waste management system. However, the torrefaction behavior is different for cotton and wipes, which are nearly pure cellulose. Torrefaction processing is also complementary to the Heat Melt Compactor (HMC) [10] as a biomass pretreatment step and is compatible with the Universal Waste Management System (UWMS) [11], both now under development by NASA. The microwave torrefaction process could also be accomplished with modest energy requirements, which could be improved even further in an optimized prototype system.

The proposed approach will make it technically feasible to process human fecal waste and related cellulosic biomass waste streams and produce additional water and other useful products in space. This will benefit long term space travel, such as an extended Lunar stay or a mission to Mars and Asteroids/Phobos. The proposed approach is also beneficial to NASA in allowing for solid waste sterilization and stabilization, planetary protection, in-situ resource utilization (ISRU) and/or production of chemical feedstocks and carbon materials. In particular, the torrefaction char residue has several potential applications in space. These include production of activated carbon, a nutrient-rich substrate for plant growth, construction material, radiation shielding, storage of elemental carbon, hydrogen and oxygen, and fuel gas (CH_4, CO, H_2) production.

Acknowledgments

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