## THERMOCHEMICAL CONVERSION OF SWINE MANURE: AN ALTERNATIVE PROCESS FOR WASTE TREATMENT AND RENEWABLE ENERGY PRODUCTION

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**ABSTRACT.** A thermochemical conversion (TCC) process was applied to the treatment of swine manure slurry for oil production and waste reduction. The objectives of the first stage study were to explore the feasibility of oil production from swine manure and to determine the waste reduction rates through the TCC process. A bench TCC reactor was developed and tested at operating temperatures of 275°C to 350°C. The corresponding operating pressures ranged from 5.5 to 18 MPa. Carbon monoxide was used as a reducing agent at pressures from 0.34 to 2.76 MPa. The oil product was evaluated by element analysis, heating value, and benzene solubility. The waste reduction rate was evaluated in terms of chemical oxygen demand (COD) before and after the TCC process. The highest oil yield was 76.2% of the total volatile solids of the feedstock. The hydrogen to carbon molar ratio was 1.53. The TCC oil product had a similar quality as that of pyrolysis oils from liquefaction of other biomass such as wood sludge and newspaper waste. The average heating value of the oil product was the sole detected gaseous by-product. The solid by-product of the TCC process was reduced as much as 75.4%. Carbon dioxide was the sole detected gaseous by-product. The solid by-product of the TCC process was only 3.3% of the total solids input by weight.

Keywords. Thermochemical conversion, Liquefaction, Swine waste, Waste utilization, Biomass.

he impact of large confinement operations of swine farms on the environment has caused increasing concerns from the general public, scientific communities, government agencies, and the pork industry. In addition, odor emission from the swine facilities has caused outcries from the public, and has become a major concern of the industry. Environmental concerns and public complaints over intensive livestock production have led state legislatures to pass new regulations on manure management, which are likely to become stringent and make pork production more costly. On top of an already slim profit margin, pork producers are now facing public complaints that could drag them into expensive legal disputes. Swine manure, once considered a nutrient-rich fertilizer, is becoming an expensive burden to the pork industry. On the other hand, swine manure is a plentiful source of biomass that has the potential to be converted into renewable energy through biological and/or chemical processes (U.S. Congress, 1980). Application of a thermochemical conversion (TCC) process to swine manure may serve dual purposes: reducing waste strength and producing renewable energy. The odor emission will also be reduced because of much shortened manure onfarm retention time.

A TCC process is a chemical reforming process in which the depolymerization and reforming reactions of ligno-cellulosic compounds occur in a heated and free oxygen-absent enclosure. The product of a TCC process depends upon the characteristics of the raw materials and the type of energy desired. TCC processes for energy production were widely studied on wood sludge, newspaper waste, and municipal sewage sludge during the oil crises of the 1970s. Among the TCC processes, direct liquefaction is the most widely studied biomass conversion process.

Direct liquefaction was historically linked to hydrogenation and other high-pressure thermal decomposition processes that employ reactive hydrogen or carbon monoxide (CO) to produce a liquid fuel from organic matter. The carbonaceous materials are converted to liquefied products through a complex sequence of changes in physical structure and chemical bonds (Chornet and Overend, 1985). Such liquefaction processes had been used to convert cellulosic wastes to oil (Appell et al., 1980). According to Appell et al., the presence of some liquid water is desirable. Elliot et al. (1988) conducted research on the direct liquefaction of several high-moisture biomass feedstocks. Kranich (1984) investigated the conversion of municipal sewage sludge to oil with carrier oil slurry and water slurry. In most of the studies, livestock waste was not included as a major biomass resource for the direct liquefaction process, although it was used as feedstock in some of the pyrolysis studies as summarized by Kreis (1979).

Swine manure is a carbon-enriched biomass. It has the potential to be converted to a liquid oil product through a TCC process. To avoid the intense energy requirement of predrying of the swine manure as in pyrolysis, it is beneficial to process the fresh manure directly without predrying. Our research explores an environmentally-friendly

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technology to treat swine manure for swine production facilities. In this study, a thermochemical conversion process, or direct liquefaction, was applied to the treatment of swine manure slurry for energy production and waste reduction. This article reports on the feasibility study of oil production and waste reduction through the TCC process of swine manure.

# MATERIALS AND METHODS TCC REACTOR

A batch mode TCC reactor was designed and developed. The TCC reactor was made of T316 stainless steel and its capacity was 1.8 L (0.5 gal). The TCC reactor could operate at extreme operation conditions of 375°C (700°F) and 34.5 MPa (5000 psi) (Parr Instruments Co., Moline, Ill.). Two agitation propellers 80 mm (3 in.) apart on a shaft were driven by a 200 W motor through a magnetic drive. Operation of the reactor was controlled by a Model 4842 controller which featured a three-term Proportional-Integral-Differential temperature control with an accuracy of  $\pm 2^{\circ}$ C (3.6°F). The temperature controller provided a maximum power of 2.5 kW to the heater. The temperature sensor was a type-J thermocouple. A high-pressure cable tubing connected the CO cylinder to the inlet of the TCC reactor for gas introduction. The reactor unit was housed in an enclosed chamber  $(2.5 \times 1.5 \times 2 \text{ m or } 8 \times 5 \times 6.7 \text{ ft})$ . An exhaust fan on top of the chamber provided a slightly negative pressure to ensure that any escaping gases from the process were exhausted outside of the operating room. The TCC reactor and its control scheme are illustrated in figure 1.

#### FEEDSTOCK

The feedstock, fresh swine manure, was collected from the partial slotted floor of a swine finisher room at the

Table 1. The statistical characteristics of feedstock swine manure

Group/Specific	Mean±S.D.
Solids content	
Total solids (TS), %	27.43±1.35
Volatile solids (VS), % of TS	87.28±1.27
Fixed solids (FS), % of TS	12.72±1.27
Elemental composition*	
Carbon, wt%	45.67±1.12
Hydrogen, %	6.45±0.21
Nitrogen, %	3.45±0.38
Sulfur, ppm	3,816±608
pH	6.06±0.17
COD, mg/L	325,200±2,000

\* Based on dry matter.

Swine Research Farm, University of Illinois at Urbana-Champaign. The fresh manure was stored at 4°C. The total solids (TS) and volatile solids (VS) contents, pH, and elemental analysis were measured. The feedstock was prepared individually for each TCC test by adjusting the total solids content with tap water to the desired TS content. The mean values and standard deviations of the feedstock characteristics are summarized in table 1.

## **PROCESS PARAMETERS**

The parameters in the TCC process include the operating temperature, carbon monoxide initial pressure, solids content, pH, and retention time. The temperature range for this study was 275~350°C. The corresponding operating pressures were 5.5~18 MPa (800~2600 psi), which was coupled with the operating temperatures under the equilibrium of water vapor and the water slurry in the operating temperature range. Carbon monoxide was used as the reducing agent in the study. The initial pressure of CO ranged from 0.34 to 2.76 MPa (50 to 400 psi). The CO initial pressure determines the initial ratio of CO to VS. The TS of the feedstock charged into the reactor was adjusted from 10% to 25% by weight. The effect of pH on



Figure 1-Diagram of the TCC reactor and control scheme.

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the fresh manure was studied at pH 4, 7, and 10. Retention time varied from 5 to 180 min for different operating conditions.

#### **PRODUCTS ANALYSES**

The TS and VS of the feedstock were measured as described in the Standard Methods for the Examinations of Water and Wastewater (Clesceri et al., 1989). Gas composition analysis was performed by gas chromatography (Model 580 by the GOW-MAG Instrument Company, Bridgewater, N.J.). The amount of gaseous product was calculated using the Starling modified BWR gas equation of state (Starling and Han, 1971). This equation was mainly used for the thermal property calculations of hydrocarbons. When tested with known  $CO_2$  data, this equation yielded a prediction with an error less than  $\pm 1\%$  with pressures up to 4 MPa. Solids analyses were performed on the solid product and post-processed water. Chemical oxygen demand (COD) was measured using a colorimetry method (HACH Co., Loveland, Colo.).

The elemental analyses, including carbon, hydrogen, nitrogen (CHN), and sulfur (S), were performed on the oil product by a carbon-hydrogen-nitrogen analyzer (Model CE440, Exeter Analytical, Inc., N. Chelmsford, Mass.) and Inductively Coupled Plasma (Plasma II, Perkin Elmer Norwalk, Conn.), respectively. Heating values of TCC oil product were estimated based on the complete oxidation of carbon and hydrogen elements and by considering the oxygen content in the oil product (He, 2000a):

$$Q (kJ/kg) = f \cdot (32\ 792 \cdot C + 142\ 900 \cdot H) + 9275 \cdot S - 2371 \cdot N$$
(1)

where C, H, S, and N are the weight fractions of carbon, hydrogen, sulfur, and nitrogen in the raw TCC oil product, respectively, and f ( $f \le 1$ ) is a correction factor of oxygen content on heating value. The results by this equation had smaller variations than those by Dulong's equation (Sawayama et al., 1996; Selvig and Gibson, 1945).

Benzene solubility and water content of the TCC oil product were performed referring to the standards for petroleum products (ASTM, 1999a,b). The benzene solubility of TCC oil product, raw oil product yield, and COD reduction rate are defined as follows:

Oil solubility = 
$$\left(1 - \frac{\text{solid residue (g)}}{\text{total oil sample (g)}}\right) \times 100\%$$
 (2)

Oil yield (%) =

$$\frac{\text{total oil product (g)}}{\text{total volatile solids input (g)}} \times 100\%$$
(3)

$$\left(1 - \frac{\text{COD of post-processed water}}{\text{COD of raw manure}}\right) \times 100\% \quad (4)$$

## **EXPERIMENTAL PROCEDURES**

The TCC reactor was operated in a batch mode. The feedstock was weighed and charged into the reactor followed by introduction of CO. The reactor was then heated at full power with a temperature increase rate of 5~10°C/min. The fastest temperature increase occurred from 220°C to 250°C, when exothermic reactions started to occur. After each run, the reactor was rapidly cooled down to 150°C or lower in 5 min by the cooling coils inside the reactor. After the reactor was further cooled to 35°C or lower, the temperature and residual pressure were recorded for gas product estimation. Agitation was set at a constant speed of 200 rpm and kept constant for all experiments. The products from this TCC process include gases, raw oil, post-processed water, and solid residues. Gas samples were vented and collected in 100 mL bottles for laboratory analysis. The raw TCC oil product had a lower density than water and floated on top of the post-processed water. It was easily separated from the rest products. After the oil product was collected, solid product and post-processed water were separated with vacuum filtration through a glass fiber filter (12 µm, HACH, Loveland, Colo.).

## RESULTS AND DISCUSSION PRODUCT DISTRIBUTION

The products after the TCC process were distributed into four different portions: raw oil product, gaseous product, solid product, and post-processed water. The amounts and composition of the different products varied according to the operating conditions. The input volatile solids distributed among all four products after the process, and the amount varied with the operating conditions. A statistical product distribution is illustrated in figure 2.

In most experimental runs, the amount of feedstock was 800 grams with water content of 80%. The CO introduction added about 1.8% to the total input. Therefore, the water content was 78.5% of the total input mass. After the TCC process, the amount of the post-processed water had a 4.4% increase compared to the water content in the feedstock. This was mainly due to the organic matter and minerals dissolved in the post-processed water.

Even though the percentages in each portion varied widely, the major portion of VS input was converted into raw oil product. Solid product separated from the post-processed water was 3.3% of the TS input and the VS in the solid product was about 7.8% of the total VS input. The portion of VS in the gas product was in the  $CO_2$  form. The above product distributions are averages based on 90 experiments. The effects of operating conditions on the TCC process, including temperature, gas initial pressure, retention time, pH and total solids content of feedstock, on the oil production and waste reduction are discussed separately (He et al., 2000b&c).

#### **TCC OIL PRODUCTION**

The conversion process of swine manure to oil is similar to other biomass liquefaction processes and to some extent it is even easier as swine manure contains less lignin and the organic matter was finely "pre-processed" by the animal digestion process. On the other hand, less lignin means less energy content and results in a lower oil yield (Humphrey, 1979; Glasser, 1985). The biomass contained



Figure 2-Product distributions: (a) Total mass balance; (b) Volatile solids balance.

in swine manure has high oxygen to carbon ratio and low hydrogen to carbon ratio (Zahn et al., 1997; Hrubant et al., 1978). These characteristics affect the oil formation efficiency negatively because high oxygen content in organic matter implies a low heating value. The swine manure slurry was completely converted into different products: raw TCC oil, post-processed water, solid residues, and gases. The conversion rate of feedstock through this process was 100%. Therefore, the conversion rate was not considered as a parameter to characterize the TCC process as in other biomass conversion processes. Addition of a reductive reagent is necessary in direct liquefaction. According to preliminary test results, little or no organic carbon was converted to oil without the addition of a reductive reagent (He et al., 1999). Temperature had a substantial effect on the oil formation. It was also observed in preliminary study that the depolymerization reactions of organic matter would not occur until the temperature reached the level where the activation energy is overcome. In this study, the preferred operating condition for successful formation of TCC oil product was 285°C to 305°C, and the corresponding operating pressures were 6.8 to 11.5 MPa (1000 to 1650 psi). These conditions were much milder compared to the reported operating conditions for other biomass studies where the operating temperature and pressure were up to 400°C and 40 MPa (5800 psi) (Elliot et al., 1988; Kranich, 1984; Appell et al., 1980). One of the notable phenomena was that at 275°C, the oil product did not form successfully in every run. About half of the experimental runs failed to yield an oil product. This was presumably because of the complexity of the swine manure composition and the slight variation from batch to batch. Representative results are summarized in table 2.

 Table 2. Representative experimental results of raw oil product

	-		-
Group	Run #1	Run #2	Run #3
Operating conditions*			
Temperature, °C	285	295	350
Pressure, MPa	7.6	9.1	18.6
Raw oil yield, % of VS	59.2	70.2	69.0
Elemental composition			
Carbon, %	71.2	73.6	77.9
Hydrogen, %	8.9	8.9	9.4
Nitrogen, %	4.1	3.9	4.6
Sulfur, ppm	0.21	0.17	0.13
Oxygen, %†	14.2	7.2	7.0
Fixed solids, % of TS	1.4	6.2	1.0
Benzene solubility, %	83.1	83.2	90.4
Heating value, kJ/kg	34,386	36,492	38,634

\* The other conditions were: RT = 120 min;  $p_{ini,CO} = 0.34$  MPa; TS = 20%; and feedstock pH = 6.1.

 $\dagger$  By difference, O = 100 - C - H - N - S - ash.

The average raw oil product yield for 90 runs was 53.8% based on the volatile solids input with a standard deviation of 19.1%. The large standard deviation is due to the variation in operating conditions. The highest raw oil product yield was 79.9% and more than two thirds of the runs achieved 50% or higher yield. The elemental composition, the benzene solubility, and heating values did not vary as much as the raw oil product yield. The average carbon and hydrogen composition in the raw oil product was  $70.8\% \pm 4.5\%$  and  $9.0\% \pm 0.5\%$ , with the highest values of 77.9% and 9.8%, respectively. The average hydrogen-to-carbon molar ratio was 1.53. The average nitrogen content in the raw oil product was 4.1% with a standard deviation of 0.4%. About 3.3% of the raw oil product was ash. The oxygen content was calculated as the difference of the other mentioned elements and ash. Its average was 11.9%. The average lower heating value (LHV) was about 34,940 kJ/kg with a standard deviation of 1590 kJ/kg. This value is lower than that of propane (49 955 kJ/kg, Lide, 1999) or heavy fuel oil (40 200 kJ/kg, Rick and Vix, 1991), but higher than those of pyrolysis oils that were 21 100~24 700 kJ/kg (Rick and Vix, 1991). Moisture content of the raw oil product ranged from 11.3% to 15.8%. Eighty out of the 90 different raw oil samples achieved a benzene solubility of 70% or higher and the highest was 96.5%. The average benzene solubility of oil product from the 80 runs was 79.5% and the distribution of the benzene solubility is as shown in figure 3. The overall properties of the raw TCC oil are similar to those oils from moist-biomass liquefaction (Elliott et al., 1988).

## WASTE STRENGTH REDUCTION

The amount of post-processed water was 82.2% of the total slurry input on average. In the post-processed water, 2.2% was volatile solids and 1.3% was fixed solids (minerals). The COD of a feedstock with 20% total solids content was 237 400  $\pm$  1200 mg/L. After the TCC process, the COD reduction was greater than 50% for all but two out of the 90 runs. The COD reduction ranged from 50% to 75%. The mean and standard deviation were 64.5% and 5.6%, respectively. A COD reduction of greater than 60% was achieved for 87% of the 90 runs (fig. 4).

It was also observed that higher COD reduction was not necessarily associated with higher raw oil yield. High operating temperatures and high CO initial pressures



Figure 3-The distribution of benzene solubility of raw oil products.



Figure 4-The distribution of COD reduction rates of 90 experiments.

favored the formation of raw oil product. However, the active CO under high temperature kept the reaction system in a reductive environment that prevented the organic and reductive inorganic compounds from oxidization, thus was unfavorable for COD reduction. If the CO initial pressure was low and the operating temperature was high, the COD reduction increased. For example, when CO initial pressure was 0.69 MPa, the COD reduction was 63% at 285°C, and 70.5% at 325°C. If the temperature remained at 285°C and the CO initial pressure increased to 2.07 MPa, the COD reduction decreased to 50%. Thus, high CO initial pressure did not benefit the COD reduction although it helped to improve the oil quality.

#### NUTRIENT CONTENTS IN POST-PROCESSED WATER

In this study, the content of nitrogen (N), phosphate (P), and potassium (K) of the post-processed water were measured for selected runs. The NPK concentrations in the post-processed water were basically constant regardless of the operation conditions, as shown in table 3. This is because the inorganic NPK from the feedstock essentially remained in aqueous solution. The major portion of the nitrogen in the feedstock was in nitrate form that dissolved in the aqueous solution. The NPK value is still too high to

 
 Table 3. Summary of the measurement results of the post-processed water

N	Р	К
(ppm)	(ppm)	(ppm)
6,300	4,196	1,130
6,300	4,330	1,471
6,300	4,062	1,200
7,300	4,089	1,352
n.d.*	4,657	938
n.d.	4,552	929
6,550	4,314	1,170
500	246	218
	6,300 7,300 n.d.* n.d. 6,550 500	6,300 4,062 7,300 4,089 n.d.* 4,657 n.d. 4,552 6,550 4,314 500 246

\* Not determined.

be discharged to a wastewater stream. Therefore, the postprocessed water needs further treatment. Research is initiated to further explore the treatment and application of the post-processed water.

#### GAS PRODUCTION AND CARBON MONOXIDE CONVERSION

Carbon dioxide  $(CO_2)$  was the solely detectable gaseous by-product in the TCC process. The GC analysis detected no methane or other gaseous hydrocarbons.  $CO_2$ production in the TCC process accounted for 20% or more of the VS input. However, the CO<sub>2</sub> did not come entirely from the VS. The carbon in the CO<sub>2</sub> was primarily from the reductive reagent CO, which combines with elemental oxygen and forms CO<sub>2</sub>, and some came from the depolymerization and decarboxylation reactions of the biomass where the carboxyl groups were thermally cleaved and the CO<sub>2</sub> was released (Chornet and Overend, 1985). It was the combination of CO with the elemental oxygen in the biomass that improved the oil product quality. Carbon monoxide consumption is an indirect indicator of oil product formation because CO eliminates elemental oxygen from the depolymerized compounds, thus yielding hydrocarbon-like products. Experimental results showed that the CO consumption ranged from 0% to 88.9%, depending on the operating conditions and CO initial addition. The average consumption was 46% based on its initial input. Although excessive CO addition resulted in a better oil yield, it did not favor the oil quality and the COD reduction when the initial CO:VS ratio exceeded 0.12 (He et al., 2000c).

## SOLID PRODUCT

Solid product was only a small portion of the total input. Based on the 90 runs under different conditions, the average solid product was about 3.3% of the total solids input. The solid product contained dirt and char, some organic matter, and a small amount of minerals. However, depending on the operating conditions, the fixed solids (FS) had a wide variation, ranging from 30% to 70%. When the oil product yield was 60% or higher, the final solid product was usually less than 1.5% of the total solids input and contained more than 75% fixed solids.

#### **ESTIMATION OF ENERGY INPUT AND OUTPUT**

Energy production is another major goal of this research. However, energy balance could not effectively and accurately be performed on this bench-scale batchmode process due to relatively large heat loss. Rough estimation of the energy input and output was conducted.

Without considering the heat loss and the energy conversion efficiency, the energy contained in the raw oil product from each experiment was 2.95 times higher than the energy needed to bring the feedstock to the desired operating temperature, i.e., 305°C. If the heat loss from the TCC system and the heat capacity of the reactor itself account for the same amount of the energy needed to bring the feedstock to operating temperature, the energy output is still about two times higher than the energy input in such a batch mode TCC process. The energy self-sustainability of the process could be more feasible if it is operated in a continuous-mode process, in which the energy content in the post-processed water could also be partially recovered and the extra energy for reactor heat capacity and heat loss can be reduced. Utilization of the TCC oil as fuel to selfsustain the process could be achieved by co-firing it with convention fuel oil. Energy balance of the TCC process of swine manure will be further investigated in future continuous-mode TCC process research.

The TCC process of swine manure has some advantages compared with other swine waste treatment methods such as anaerobic digestion. Anaerobic digestion is a common practice of waste reduction and renewable energy production from swine manure. It converts about 50% of the organic matter into biogas, composed mainly of methane and CO<sub>2</sub>, (U.S. Congress, 1980). However, the retention time in anaerobic digestion is long, thus large digesters are needed for large confinement swine farms. The digested sludge (about 50% of the total input) needs to be further treated too. The TCC process converts organic matter in the raw manure completely. It could directly process fresh manure from a barn and storage requirement is substantially reduced. The TCC reactor is compact and takes much less space than an anaerobic digester for the same size swine facility. The TCC process is a closed system and, combining with short manure storage time, the odor emissions from the TCC process can be greatly reduced. The post-processed water contains no settleable solids, making the water suitable for further treatment. Because a large portion of the organic matter is converted to oil product through such a thermochemcial process, solid residues are minimized (about 3% of the total input) and panthogen-free. The oil product from the TCC process is a higher quality energy form and easier to collect, transport, store, and utilize than the biogas. One of the limitations of the TCC process is its intense capital cost and operating cost. However, if the oil product is effectively used as fuel to self-sustain the operating energy consumption and/or as high value added feedstock for valuable chemical products, the return from such a renewable production process could be high. As the environmental regulations are getting more stringent, the TCC process could be a sustainable technology for treating swine manure.

## CONCLUSIONS

The TCC process was successfully applied to the treatment of swine manure slurry to produce liquid oil and reduce the waste strength. The oil yield was as high as 76.2% of the total volatile solids of the feedstock and the hydrogen to carbon molar ratio was 1.53. The TCC oil product had a similar quality to that of pyrolysis oils from

liquefaction of other biomass. The average low heating value of the oil product was estimated as 34 940 kJ/kg. The COD in the post-processed water after the TCC process had been reduced as much as 75.4%. Carbon dioxide was the sole gaseous by-product that accounted for 16.9% of the total volatile solids of the feedstock. The solid product of the TCC process was only 3.3% of the total solids input in feedstock. In summary, the TCC process can be potentially applied to the treatment of swine manure. It can not only reduce the waste strength, but also produce useful energy in the form of liquid fuel.

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