

SWINE MANURE SOLIDS SEPARATION AND THERMOCHEMICAL CONVERSION TO HEAVY OIL

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Separation of solids from liquid swine manure and subsequent thermochemical conversion (TCC) of the solids fraction into oil is one way of reducing the waste strength and odor emission. Such processing also provides a potential means of producing renewable energy from animal wastes. Gravity settling and mechanical separation techniques, by means of a centrifuge and belt press, were used to remove the solids from liquid swine manure. The solid fractions from the above separation processes were used as the feedstock for the TCC process for oil production. Experiments were conducted in a batch reactor with a steady temperature 305 °C, and the corresponding pressure was 10.34 Mpa. Gravity settling was demonstrated to be capable of increasing the total solids content of manure from 1% to 9%. Both of the mechanical separation systems were able to produce solids with dry matter around 18% for manure, with 1% to 2% initial total solids. A significant amount of volatile solid (75.7%) was also obtained from the liquid fraction using the belt press process. The oil yields of shallow pit manure solids and deep pit manure solids with belt press separation were 28.72% and 29.8% of the total volatile solids, respectively. There was no visible oil product obtained from the deep pit manure solids with centrifuge separation. It is believed that it is the volatile solid content and the other components in the manure chemical composition which mainly determine the oil production.

Keywords: Thermochemical conversion; Separation; Manure; Oil

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INTRODUCTION

With the expansion in livestock industries, proper management of animal wastes has become an economic and environmental imperative. Liquid manure systems are popular among livestock and poultry operations in the United States, Canada, and Europe because they make manure easy to handle, store, and treat in anaerobic lagoons, storage tanks or in pits below slotted floors. However, the odor generation from lagoons and storage structures has increased public concern over the use of liquid manure storage systems (Chastain et al. 2001). Solids-liquid separation has traditionally been used as a primary treatment process to improve liquid manure handling properties and generate solids for composting, energy generation, and re-feeding to animals (Zhang and Westerman 1997). Effective solids and liquid separation can remove substantial organic

matter from manure, offering the potential of producing nutrient-rich organic solid, and odor reduction in subsequent manure treatment and storage structures (Zhang and Westerman 1997).

Several methods are available to separate solids from liquids, including sedimentation (solids settle by gravity), mechanical separation (screen separators, centrifuge, hydrocyclones, screw press, and belt press), evaporation ponds, dehydration, coagulation, and flocculation (Zhang and Westerman 1997). Solids-liquid separation via gravity settling has been used extensively to reduce the solids content in feedlot runoff and flushed dairy manure (Chastain et al., 2001). Gravity settling of swine manure can remove as much as 60% of total solids from swine manure (Lorimore et al. 1995). However, the separated solids have high water content and must be dewatered for further uses, such as composting. Few studies have reported on mechanical separation of flushed swine manure. It was reported that belt presses and centrifuges have higher separation efficiencies and produce drier solids than the screen separators (Zhang and Westerman 1997). A centrifuge can be used to produce swine solids with a dry matter content ranging from 16% to 27% (Piccinini and Cortellini 1987). A belt press can be used to remove as much as 59% of the TS with a dry matter content up to 18% (Fernandes et al. 1988).

Separated solids can be used for composing, refeeding, or generating biogas (methane) (Worley and Das 2000; Demirer and Chen 2005), and one of these uses may be thermochemical conversion of these solids into oil products. Thermochemical conversion (TCC) is a chemical reforming process of organic compounds and water in a heated enclosure to produce oil and gas. The products of the TCC process are dependent on the characteristics of the raw material and the process operating parameters.

Research on thermochemical conversion of biomass into renewable energy began in 1970s. Since then, TCC processes for energy production have been widely studied for high lignin and cellulose content biomass such as wood (Demirbaş 2005; Garrote et al. 1999; Minowa et al. 1998; Kruse and Gawlik 2003). There has been little work in the TCC of biomass with low contents of lignin and cellulose, especially animal waste (Itoh et al. 1994; Raymond and Kieffer, 1995; Williams and Besler 1992). Recently, the University of Illinois research group has successfully developed a TCC process to convert fresh swine manure into oil product (He et al. 2000a, 2000b; Zhang et al. 2004). In this process, the fresh manure is treated during 20-120 min with water under subcritical conditions (275-350 °C, 5.5-18Mpa) to give a heavy organic oil (biocrude) with a heating value of 30-35MJ/kg. This earlier work showed that the technology has potential to treat manure effectively and can be used as a method to produce a liquid biofuel (Ocfemia et al. 2006). However, no study has been carried out in terms of TCC processing of liquid manure. In the present work, liquid swine manure was separated by different methods, and the subsequent solids fraction was converted into oil by TCC processing.

The purpose of this research was to figure out a practical and economical way of separating liquid swine manure and getting a high solid content solid for the TCC process, and to investigate the feasibility of oil production from separated solids through the TCC process. This is the first time that the TCC technique has been used to convert liquid swine waste - an abundant, costless feedstock - into bio-oil. The feasibility study

provides essential information on the effect of the separation method and swine manure sources on the bio-oil production by using the TCC process. Thus, the research can provide important information on the commercial utilization of liquid manure for renewable energy production.

MATERIALS AND METHODS

Solids-liquid Separation

The raw materials for the solids-liquid separation test were shallow pit manure and deep pit manures. The shallow pit manure was taken from the pits below slotted floors in Grein's Farm on the South Farms at the University of Illinois. The deep pit manures were provided by World Wide Bio Energy Company, which were taken from one anaerobic lagoon in Jefferson City. The oil production would not be very effective if the TCC process handled a feedstock with a low total solids (He 2000c). Therefore, in an attempt to achieve a solid content of approximately 20%, which is desired for TCC process, these liquid manures were separated by settling tank, centrifuge, and belt press, respectively. The solids fraction obtained from running these pit manures through these separation operations were used as the feedstock for the TCC process.

A gravity settling tank was developed to remove solids from the liquid manure (Fig. 1). The tank used was approximately 1.27 meters long and 0.30 meters in diameter. There were eight sampling outlets installed along the length of the tank, ranging from the bottom to the top. After settling, the samples could be taken out from each point for solid content analysis. A basket centrifuge and a belt press were used as alternative methods to separate the deep pit manure.

In order to determine how well each of the separation methods would work and to answer the question which separation method would best fit the process for converting pit manure to oil, the performance of settling tank and mechanical separators was evaluated by the moisture content of solids separated. A low moisture content of the solids separated is desirable.

After each separation, the solid fractions were sampled and analyzed for further TCC study. These analyses included solids content analysis, forage analysis (crude protein, crude fat, crude fiber, starch), detergent fiber analysis (cellulose, hemicellulose, lignin), and minerals (S, Ca, Mg, Na, Cu, Fe, Mn, Zn) content analysis. All analyses were conducted by the Midwest Laboratories Inc., a private laboratory located in Omaha, Nebraska. The forage analysis and detergent fiber analysis methods were in accordance with the National Forage Testing Association (NFTA) guidelines. Mineral content was determined using wet digest procedure performed by Inductively Coupled Argon Plasma (ICAP/ICP).

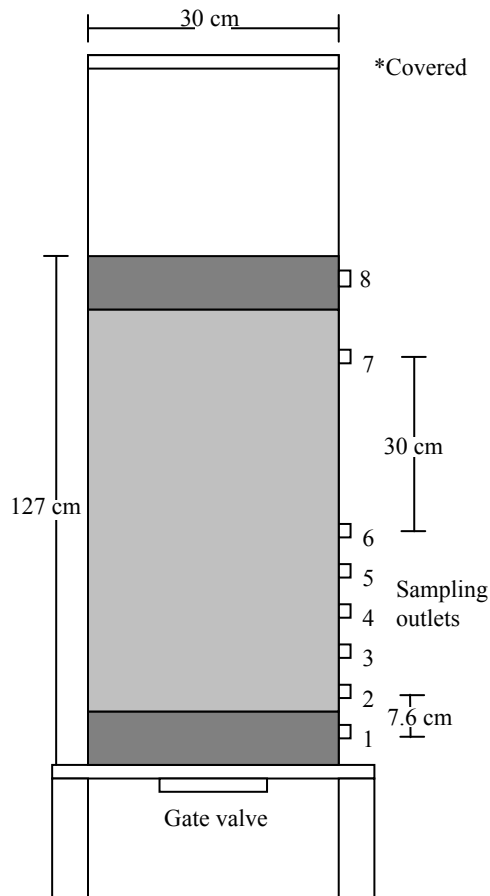


Fig. 1. Schematic diagram of the settling tank

The solids content analysis was determined using the APHA-AWWA-WPCF Standard Method 2540, which includes total solids (TS), volatile solids (VS), and fixed solids (FS). The total solids are the residues remaining in a vessel after evaporation in a drying oven. The Fixed solid is the remaining solids in a crucible after incinerating the dried solids, while the weight lost on ignition is the volatile solids. In this procedure, the samples were dried completely by holding them in a drying oven for 24 h at a temperature of 105°C. The mass of TS was determined after the samples were allowed to cool in a desiccator. The mass of FS were determined by incinerating the dried solids in a furnace that was maintained at 600°C for 1.5 to 2 h, and allowing the samples to cool in a desiccator. The mass of VS was simply the difference between the total and fixed solids. The TS, VS, and FS can be calculated as follows,

$$\text{Total solids (\%)} = \frac{(A - B) \text{ (g)}}{\text{wet sample weight (g)}} \times 100\% \quad (1)$$

$$\text{Volatile solids (\%)} = \frac{(C - D) (\text{g})}{\text{Dried sample weight (g)}} \times 100\% \quad (2)$$

$$\text{Fixed solids (\%)} = \frac{(D - B) (\text{g})}{\text{Dried sample weight (g)}} \times 100\% \quad (3)$$

where:

A = weight of residue + dish after drying, g

B = weight of dish, g,

C = weight of residue + dish before ignition, mg, and

D = weight of residue + dish after ignition, mg,

Thermochemical Conversion

The TCC experiments for these swine manure solids were carried out in a 1.8 L high-pressure high-temperature stainless steel reactor. It is rated up to a working pressure of 20.68 Mpa and working temperature of 350°C. A heavy-duty magnetic drive stirrer is installed for mixing. A type-J thermocouple is fitted into the reactor for direct temperature measurements of the reaction media. A standard pressure gauge is installed on the reactor head. A PID controller is used to control the temperature of the reactor. An electric heater provides heating (i.e. on the outer wall of the reactor vessel).

The operating conditions that were used in the tests were subjected to the TCC process at optimal operating conditions found for fresh swine manure. The conditions were as follows: temperature, 305°C; retention time, 60 min; operating pressure, ~10.34 Mpa; process gas (N₂) with 0.69 Mpa initial pressure.

In a typical TCC experiment, the reactor was loaded with 600 ml of manure. Then the reactor was purged 3 times with nitrogen to remove the air/oxygen in the reactor airspace. Agitation was set at 200 rpm and kept constant for all experiments. The temperature was then raised up to 305°C and kept for 60 min at 305 °C. The temperature and pressure were monitored and logged continuously. After reaction, the reactor was cooled down to the room temperature by means of a cooling coil, which was installed inside of the reactor. The temperature and pressure were monitored and recorded every 5 min. The heating rate and cooling rate were around 7°C/min and 10 °C/min, respectively.

The products from the TCC process included gases, raw oil, post-processed water, and solid residues. The gaseous products were vented. The raw TCC oil product had a lower density than water and floated to the top of the liquids. The solid and liquid products were separated by filtration under vacuum through a glass fiber filter (12 μm, HACH, Loveland, Colo.). In this study, the term “oil product” was defined as the fraction that floated on the top of the aqueous phase. This definition is different from that used in some other studies. For example, Ogi et al. (1985) referred to the fraction that was collected and separated from the dark brown or viscous material as the oil products. It was not reported whether they observed any oil product floating on the top of the reactor contents.

Oil production was used to measure the TCC efficiency. Benzene solubility of the oil product was used as an indicator of oil product quality. The oil yield represents the amount of organic material in the manure that has been converted into oil. The higher the

benzene solubility of oil products, the higher the oil quality. The raw oil product yield and the benzene solubility of TCC oil product are defined as follows:

$$\text{Oil yield (\%)} = \frac{\text{total oil product (g)}}{\text{total volatile solids input (g)}} \times 100\% \quad (4)$$

$$\text{Oil solubility (\%)} = \left(1 - \frac{\text{solid residue(g)}}{\text{total oil sample(g)}}\right) \times 100\% \quad (5)$$

RESULTS AND DISCUSSION

Solids Separation

There were no observable distinct layers formed in the settling tank after an hour. After 24 hours, 3 distinct layers were formed: a bottom layer with settled solids (see Fig. 1), a middle layer with suspended solids, and a top layer with light solids and some foam. Corn material was found to be on both bottom and top layers. Some solids on top may have been brought up by light flocculated material. Disturbing (by tapping) the container caused some particles to settle from the top layer.

Attempts were also made to use the settling tank to separate the deep pit manure. But this quickly proved ineffective and not successful, since there was no evidence of distinct layers in the settling tank after 48 hours.

Samples were taken from each of the sampling outlets (see Fig.1) to determine solids content at various locations. Figure 2 shows the solid analysis results of each sampling outlet for shallow pit manure and deep pit manure. In the case of shallow pit manure, instead of most solids settling to the bottom layer, the highest solids content was obtained from the top layer (point 8), which was up to 29.17%. The solids contents at outlet points 2, 4, 6, and 7 were very similar to each other, with values ranging from about 0.87 to 1.01%.

Considering that these settling points on the middle part of the container had low solids content, samples of the slurry from the top and the bottom were collected as components of the test samples. After mixing the two parts homogeneously, the solids content of the mixed manure was also analyzed, which was about 9%. Since the solids content of separated solids was still much lower than 20%, additional water was evaporated in the oven at 60 °C for 9 hours, and finally 20% solids content was obtained. In the case of deep pit manure, no significant differences between each sampling outlet were observed, indicating that this kind of manure cannot be separated effectively by a settling tank. The characteristic parameters of animal manure that affect the solid-liquid separation performance mainly include particle size distribution and initial total solids content (Zhang and Westerman 1997).

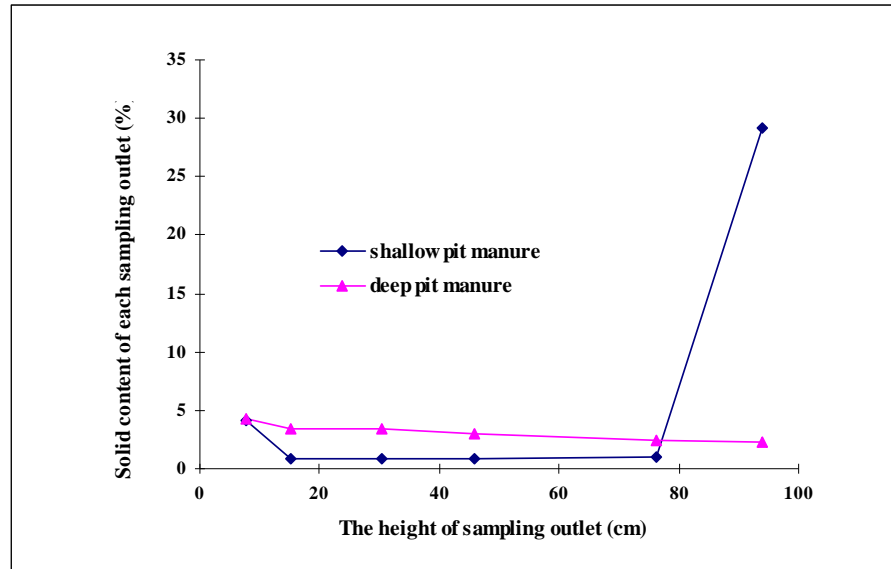


Fig. 2. The solid content analysis results at different sampling outlets for shallow pit manure and lagoon pit manure

For comparison of the solids content of in and out samples through centrifugal and belt press processes, the total solid (TS) and volatile solid (VS) concentration of the raw deep pit manure and the solid fraction after separation are shown in Fig. 3 and Fig. 4, respectively. For the belt press process, the TS content increased from 0.8% to 17.7%, and the VS content increased from 46.2% to 75.7%. The centrifugal separation process increased TS content of deep manure from 1.84% to 17.17%, but it did not show any significant difference in the VS content between the in and out samples.

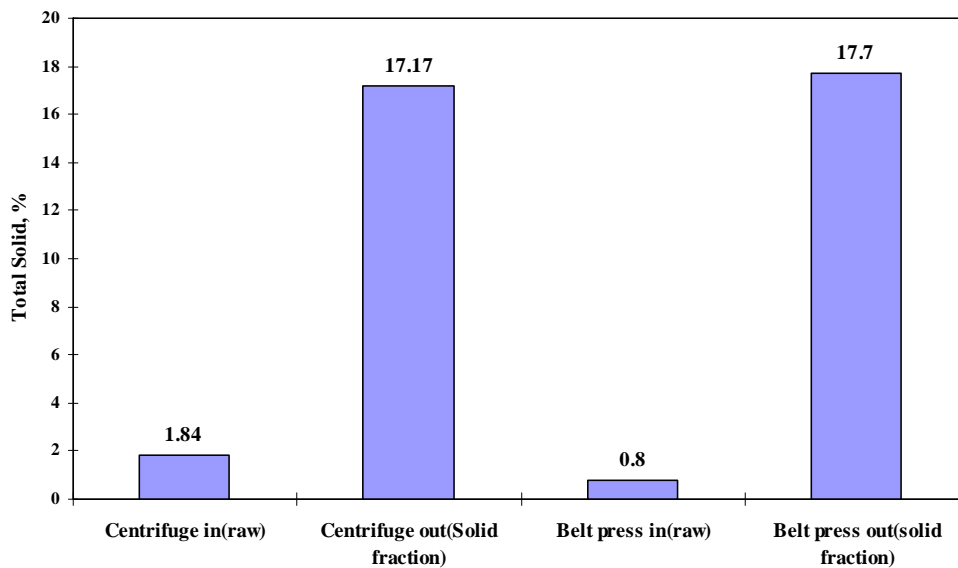


Fig. 3. Total solids (TS) content for in and out samples for the centrifuge and belt press separation

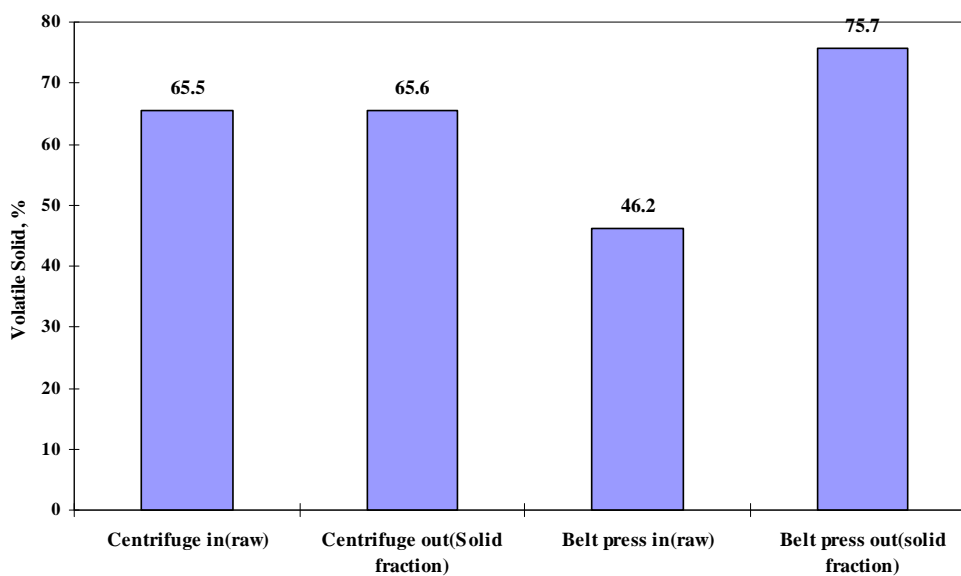


Fig. 4. Volatile solids (VS) content for in and out samples for the centrifuge and belt press separation

Solid Analysis

The compositions of samples of the separated swine manure solids are listed in Table 1. The composition analysis results for fresh manure are also reported in Table 1 for comparative purposes. One of the most notable differences between the pit manures and fresh swine manure is the significantly higher ash content of pit manure, especially for deep pit manure with centrifugal separation. The ash content of this kind of manure reached 34.4%, which corresponds to a very lower VS content, with the value of 65.6%. Differences also existed in the carbohydrates group among these samples. For example, the lignin content was a lot higher (up to 14%) in deep pit manure with centrifugal separation, compared to the other manures. Deep pit manure with belt press showed considerably more cellulose and crude fat than the other manures. The mineral content of the fresh manure and shallow pit manure were not strikingly different, except for some components. Comparing with fresh manure and shallow pit manure, deep pit manure had a lower mineral content for most of the elements, including the alkali metal and crude protein content. These components are believed to be the catalysts in the reaction for conversion to oil (Oji et al. 1985; Inoue et al. 1999).

Table 1. Characteristics of Fresh Manure, Shallow Pit, and Deep Pit Manure

No.	Manure 1	Manure 2	Manure 3	Manure 4
Group/specific	Fresh	Shallow pit	Deep pit	Deep pit
Collecting method	From floor	Gravity Settling	Centrifuge	Belt press
<u>Solids content</u>				
Total Solid, %wt	27.43	17.90	17.91	17.7
Volatile solids, % dry matter	86.60	79.60	65.60	75.70
Ash, % dry matter	12.30	20.40	34.40	24.30
H ₂ O, %wt	72.57	82.10	82.08	82.30
Σ	100	100	100	100
<u>Compositions of dry matter %wt</u>				
hemicellulose	20.50	22.25	18.70	35.10
cellulose	8.20	5.70	15.20	27.68
Lignin	3.10	2.65	13.40	4.22
ash (%)	12.30	20.40	34.40	24.30
crude fat (%)	7.46	22.70	5.95	27.00
Crude protein(Kjeldahl)	29.30	21.20	17.90	29.30
<u>Element group(%)</u>				
Sulfur(S)	0.42	0.73	0.10	0.12
Phosphorus (P ₄ O ₁₀)	1.71	2.94	0.88	1.83
Potassium (K ₂ O)	2.37	2.70	0.63	0.11
Magnesium (Mg)	0.71	1.18	0.19	0.28
Calcium (Ca)	0.33	3.66	0.28	1.58
Sodium (Na)	1.90	0.51	0.13	0.02
Iron(ppm) (Fe)	1,708	2,733	347	1,125
Manganese(ppm) (Mn)	244	352	73	139
Copper(ppm) (Cu)	100	139	36	43
Zinc(ppm) (Zn)	689	1231	177	143

TCC Test

Oil product was obtained from shallow pit manure and deep pit manure with belt press separation. No visible oil was found from deep pit manure with centrifugal separation. In the case of shallow pit manure, the products after the TCC process were distributed into four different portions: raw oil product, aqueous product, gaseous product, and solid product. The raw oil product floated on the top of the reactor vessel, the aqueous product was in the middle and the solid product settled on the bottom of the reactor.

In the case of deep pit manure solid after belt press separation, the oil product, which contained dirt, char, and some heavy oil, floated on the top, and some of these components were attached on the inner wall of the reactor. The solid and liquid could be easily separated by filtration. In the case of the deep pit manure solid after centrifugal separation, no observable oil floated on the top. After the test, only aqueous product was obtained, which was dark in color, almost black. Some solids settled immediately and were black in color. There might have been some oil combined with the solids, but the amount of the oil was too small to be observed.

The results of oil yield and benzene solubility of oil product are shown in Fig. 5. In comparison with fresh manure, at the same operating conditions, both shallow pit manure and deep pit manure with belt press produced a lower oil yield. The oil yields of

shallow pit manure solids and deep pit manure solids with belt press separation were 28.72% and 29.8% of the total volatile solids, respectively. And moreover, the benzene solubility of both of oil products was also lower than fresh manure. The benzene solubility of oil produced from deep pit manure with belt press separation was quite low with value of 29.2%, indicating a bad oil quality.

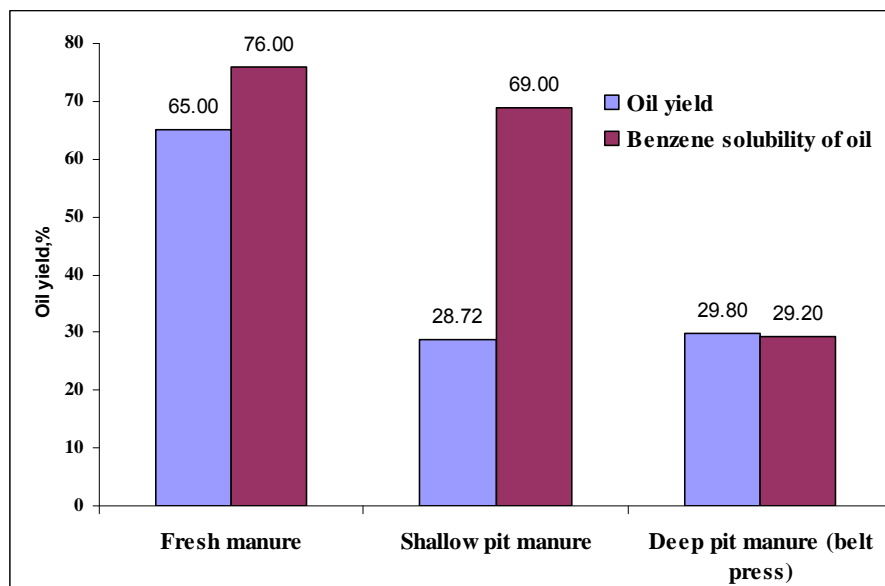


Fig. 5. Oil yield and benzene solubility to pit manure and fresh manure by TCC process

The difference in composition may explain why crude oil could be produced from fresh manure, shallow pit manure, and deep pit manure with belt press separation, yet not from deep pit manure with centrifugal separation. As described in the solid analysis section, it was noticed that the deep pit manure with centrifugal separation contained higher ash and lignin content, and lower total volatile solid, crude fat, and crude protein content.

The VS is a measure of the amount of organic matter present in the solid fraction, which is the portion having the greatest potential to be converted into flammable products. More VS is desirable for the purpose of oil production. On the basis of 20% total solids, a smaller fraction of the total solids in the pit manure could be converted to oil because the amount of organics was significantly lower as compared to that of fresh swine manure. In addition, our previous tests on evaluating the effect of solids content to oil yield show that there was a sharp decline in oil yield when the VS was below 87%wt of total solids (He 2000c). Therefore, it is reasonable to assume that there exists a critical minimum VS content to produce oil. The value of the critical minimum VS is expected to be further determined with a carefully laid out experimental design.

It's believed that any organic material, specifically carbohydrates, proteins, and fats could be converted into crude oil (Kruse and Gawlki 2003; Minowa et al. 1998). Suzuki et al. (1988) found that a nearly linear relationship exists between crude fat content and amount of oil in the sewage sludge. Recently, Demirbaş et al. (2005) reported

that the oil yields were lower for those woods that contained a larger amount of lignin. This is in agreement with our current study.

The fact that there was no oil produced from deep pit manure with centrifugal separation may be attributed to the water-soluble components in pit manure. Some of the water-soluble components may act as the critical factors that could lead to the formation of oil; however they could not be recovered by the centrifugal separation process. However, other factors, such as interactions between the component fractions, catalytic actions by the mineral matter content, and inhibitors that could prevent the formation of oil, also may play a role in the formation of oil and oil yield. These are some of the parameters that need to be further investigated in order to better understand the reactions involved in the process. A long-term study would be necessary to get an insight into the reaction mechanism and kinetics for the thermochemical conversion of animal waste and other low cellulosic wastes.

CONCLUSIONS

Gravity settling was able to increase TS content of shallow pit manure from 1% to 9%, while it proved ineffective for separating the deep pit manure, since there were no evidently distinct layers formed in the settling tank after 48 hours, indicating that this kind of manure is hard to separate by gravity. The belt press and centrifuge were found to have higher separation efficiencies and produce drier solids than the settling tank. The separated solids generated from the two separators contained high solid content up to 18% and did not need to be further dewatered for the TCC process. In comparison with the centrifugal process, the belt press proved to be more efficient to reduce the volatile solids loading rate to the lagoon. In the other words, a belt press can trap more volatile solids staying in the solids fraction, which would have more contribution to the oil generation.

The separated solids fraction generated from shallow pit manure and deep pit manure were tested in a batch reactor through the TCC process. It was found that oil could be produced from shallow pit manure and deep pit manure with belt press separation. In comparison with fresh manure, at the same operating conditions, both shallow pit manure and deep pit manure with belt press separation produced a lower oil yield. No visible oil was found for deep pit manure with centrifugal separation. Comparative composition analysis showed that the oil yield was highly dependent on the composition of the starting material, such as volatile solid, lignin, crude fat, and water-soluble compounds in the manure.

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