

# COMBUSTION FUEL PROPERTIES OF MANURE AND COMPOST FROM PAVED AND UNPAVED CATTLE FEEDLOTS AS MODIFIED BY ANNUAL PRECIPITATION

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**ABSTRACT.** Research was conducted to determine the effects of feedlot surfacing materials (soil/unpaved vs. fly-ash paved), partial composting and/or storage in windrow or greenhouse, and wet year (2005) vs. dry year (2006) feedlot conditions on cattle feedlot biomass (FB) characteristics pertinent to combustion or gasification processes involving reburn or co-firing with coal or lignite as described elsewhere. Bulk quantities of FB were harvested from 12 fly-ash paved pens and six soil-surfaced pens in a 400-head research cattle feedlot near Bushland, Texas. FB was windrow-composted, and samples were analyzed using proximate, ultimate, and elemental ash analyses. Higher heating value (HHV) before composting (termed raw manure, RM) was significantly higher for FB harvested from paved pens (termed low ash, or LA-FB) vs. soil-surfaced pens (termed high ash, or HA-FB) on a dry basis and dry ash-free (DAF) basis for both study years. Ash content (dry matter basis) was significantly lower and volatile matter (VM), carbon (fixed and total), and hydrogen were significantly higher for LA-FB (paved pens) vs. HA-FB (unpaved pens) for both years of the study. As-harvested HHV averaged 6,304 and 8,190 kJ kg<sup>-1</sup> w.b. (2,710 and 3,521 BTU lb<sup>-1</sup> w.b.) for HA-FB for the wet and dry years, respectively, and as-harvested HHV for LA-FB averaged 13,407 and 13,224 kJ kg<sup>-1</sup> w.b. (5,764 and 5,685 BTU lb<sup>-1</sup> w.b.) for the wet and dry years. Partial composting (PC) in windrows (for 51 to 55 days in 2005 or 91 days in 2006) reduced HHV w.b. by an average (both years) of 11.4% for the resulting HA-FB-PC and 14.8% for the resulting LA-FB-PC. The LA-FB-PC was significantly different from HA-FB-PC for all proximate and ultimate analysis parameters for both 2005 and 2006 manure, with the exception of 2006 wet-basis HHV. Heating value on a DAF basis averaged 20,984 kJ kg<sup>-1</sup> (9,022 BTU lb<sup>-1</sup>) for LA-FB-PC and 18,941 kJ kg<sup>-1</sup> (8,143 BTU lb<sup>-1</sup>) for HA-FB-PC. Greenhouse storage for 9 to 12 months of the 2005 harvested LA-FB-RM and LA-FB-PC preserved fuel quality better than did continuous storage of LA-FB-PC in a windrow, probably because lower moisture conditions were maintained under greenhouse storage conditions (7.7% to 13.4% w.b. final range) compared with uncovered windrow storage (17.4% final moisture content). Elemental analysis of ash from LA-FB was higher than from HA-FB for Ca, P, Cl, K, Mg, Na, and S, but was lower for Si, Al, Ti, and Fe without or with partial composting. Metal contents were similar for both high-ash and low-ash FB.

**Keywords.** Air quality, Beef cattle, Biomass, Livestock waste, Manure, Renewable energy.

The Texas High Plains is considered the center of the nation's cattle feeding industry, with 42% of the U.S. fed beef production within a 322 km (200 mi) radius of Amarillo, Texas, including Texas and the neighboring states of Oklahoma, New Mexico, Kansas, and Colorado. Manure produced from the 7.2 million head fed each year amounts to more than 4.5 million metric tons (5 million tons) per year on an as-

collected basis. Heretofore, this harvested feedlot manure has been used extensively for irrigated or dryland crop production or on pastures in the general vicinity. Over half the grain fed in area feedlots is imported from out of state (e.g., Midwest). Declining water tables in the Ogallala Aquifer in the Texas High Plains, withdrawal rules adopted by regional groundwater conservation districts, and increasing fuel costs have reduced irrigation water use per acre. As these trends continue, future demands for manure as fertilizer on a per-acre basis could decline, such that feedlots might encounter longer hauling distances to achieve P-based or N-based nutrient balances on irrigated or dryland cropping systems. The region also supports a rapidly growing dairy production industry that experienced a 10-fold expansion in the decade of 2000 to 2010. This growing dairy production adds to the available manure supply. New manure management approaches are becoming necessary for a sustainable beef cattle feeding industry in this region. Innovative technology and multi-media environmental approaches to manure management that conjunctively address water and air quality, soil quality, energy usage, climate change, and biomass energy utilization are needed to meet

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future policies (Auvermann and Sweeten, 2005).

Energy use at cattle feeding operations is substantial (Sweeten, 1996), and costs continue to escalate. Potential exists for on-site production and utilization of renewable energy, including biomass conversion (Annamalai et al., 2007). Renewable energy options involving animal wastes include: (1) methane capture from anaerobic waste storage/treatment units, and (2) thermochemical conversion using pyrolysis, combustion (including co-firing with coal or lignite) (Annamalai et al., 2003; Arumugam et al., 2005b), gasification (Priyadarsan et al., 2004, 2005), or reburn processes (Arumugam et al., 2005a; Annamalai et al., 2005). Thermochemical conversion greatly reduces the volume of volatile materials. The resulting combustion residue (ash) contains noncombustible minerals including N, K, P, and Cl, which could be transported greater distances than bulk manure, provided that these materials can be utilized beneficially. Thermochemical conversion may also provide a means of utilizing composted carcasses that could result from normal or even catastrophic mortalities on a local or regional scale (Auvermann and Sweeten, 2005).

A main focus of the investigators has been on maximizing higher heating value (HHV), minimizing ash content, and identifying potential constraints of mineral contaminants (S, Cl, Na, K, P, etc.) of feedlot biomass (FB) that can contribute to ash agglomeration or slagging in combustion units (Sweeten et al., 2003). The specific uses of the FB material and subsequent data collected were to support further research by multiple investigators concerning:

- Reburn technology to reduce emissions of nitrogen oxide (NO<sub>x</sub>) (Annamalai and Sweeten, 2005) and mercury (Hg).
- Preparing, characterizing, and supplying manure from the Texas A&M AgriLife Research/USDA-ARS experimental feedlot at Bushland, Texas, or from commercial feedlots, to specification for use in combustion, gasification, and/or reburn experiments to be conducted in a 29.3 kW (100,000 BTU h<sup>-1</sup>) pilot facility in the TAMU Mechanical Engineering Department Renewable Energy Laboratory, Texas A&M Engineering Experiment Station (TEES).
- Design of a manure-fired ethanol production plant near Hereford, Texas, by Panda Energy Group.
- Agronomic uses for ash generated from thermochemical conversion technologies using FB (Megel, 2007).
- Evaluating important combustion properties, including chemical characteristics of the FB bio-fuels prior to combustion or gasification experiments (Annamalai et al., 2012; Sweeten et al., 2012).

## OBJECTIVES

The purpose of this research was to evaluate feedlot biomass as a renewable energy resource for thermochemical conversion processes, including combustion or gasification. Specific objectives were to: (1) characterize harvested cattle feedlot manure from paved vs. unpaved feedpens as a biomass energy feedstock, and (2) determine differences in harvested feedlot biomass chemical characteristics and heating value as a function of feedlot surfacing materials,

antecedent precipitation, partial composting, and storage in a greenhouse or windrow.

The primary research focus was on determining open-lot manure feedlot biomass (FB) characteristics as a biofuel feedstock as a precursor to its preparation and use in biomass energy conversion systems involving reburn or co-firing with coal or lignite as base fuel. Ultimate, proximate, and ash analyses were performed on FB as well as on Wyoming Powder River basin (PRB) coal and Texas lignite for subsequent pilot plant experiments reported elsewhere.

## MATERIALS AND METHODS

### FEEDLOT BIOMASS SOURCE AND HARVESTING

The source of FB used in this intensive characterization project came from 18 feedpens of eight head of beef cattle each in the 400-head Texas A&M AgriLife Research/USDA-ARS research feedlot near Bushland, Texas. The FB resulted from two successive 135-day to 150-day beef cattle feeding trials, which were concluded at the end of May 2005 and May 2006. When the feeding trials were terminated, the FB was harvested using a skid-steer wheel loader from 12 feedpens that were paved in 1998 with 15 to 20 cm (6 to 8 in.) depth of hydrated, graded, and compacted mixtures of crushed bottom ash and fly ash from the Harrington Station coal-fired power plant near Amarillo, Texas. These paved pens provided a stable hardened pen surface that prevented entrainment of fly ash material, or underlying soil, with the deposited and harvested manure. The resulting manure removed from this pen surface was termed low-ash feedlot biomass (LA-FB). The six remaining eight-head pens were unpaved, soil (Pullman clay loam) surfaced pens, which are typical in the Southern Great Plains cattle feeding industry. The resulting manure removed from the unpaved pens was termed high-ash feedlot biomass (HA-FB) in that there was no preventative mechanism to restrict entrainment of the deposited manure with the underlying soil either by cattle hooves or harvesting machinery. This soil entrainment/contamination is the primary reason that ash content in manure from soil-surfaced pens is higher than from paved pens.

FB was removed, stockpiled, and began partially composting on 9 or 13 June 2005 following the winter-spring 2005 cattle feeding trials, which were conducted during a prolonged wet winter and spring. Because of antecedent wet weather conditions, i.e., 38.4 cm (15.1 in.) of precipitation in the preceding nine months (September 2004 to May 2005), there was maximum opportunity for entrainment of underlying soil from hoof action in the unpaved feedpens. The 12 paved feed pens harvested on 20 May to 8 June 2005 produced an average of 3,212 kg (7,083 lbs) FB per pen as collected, which was termed low-ash feedlot biomass (LA-FB). The six soil-surfaced (unpaved) feedpens harvested on 1 to 10 June 2005 yielded an average of 4,233 kg (9,333 lbs) FB per pen as collected, which was termed high-ash feedlot biomass (HA-FB) in reference to its higher entrained soil content. The as-collected LA-FB and HA-FB were placed into separate windrows for sampling prior to the start of composting.

## PREPARATION AND SAMPLING PROTOCOLS FOR MANURE AND COMPOST

FB samples used to obtain fuel characteristics were taken after FB harvesting and prior to composting, after composting, and/or after long-term storage. Samples prior to composting were termed raw manure (RM), and samples taken after 51 to 91 days of composting, involving successive wetting and turning cycles, were termed partially composted (PC). Samples taken after longer-term storage (9 to 12 months) were additionally termed windrow-stored (WR) or greenhouse-stored (GH). Three composite 2 kg samples ( $n = 3$ ) were extracted from ten subsamples randomly collected from each type of FB material: LA-FB-RM, HA-FB-RM, LA-FB-PC, HA-FB-PC, greenhouse-stored, and windrow-stored.

In addition, prior to the start of composting, bulk samples of the freshly harvested manure were extracted from each windrow using the skid-steer loader, with composite samples of ten subsamples each totaling 953 or 318 kg (2,100 or 700 lbs) of LA-FB or HA-FB, respectively (where the terms LA-FB or HA-FB are used without the RM or PC modifiers, they imply RM for the purposes of this article). These bulk samples of FB were coarsely ground in a small hammer mill and placed in a greenhouse (2 to 10 June 2005) to facilitate drying to  $\leq 10\%$  moisture by mass wet basis (w.b.). Again, three 2 kg composite samples comprised of ten subsamples each were extracted after grinding and prior to placement in the greenhouse, and these were submitted for analysis also.

## ANALYSES

The FB materials collected and processed were analyzed in a commercial laboratory to determine their composition with respect to potential biofuel application. Proximate, ultimate, and ash analyses of the fuels were conducted, and the results were used to plan and conduct subsequent gasification, combustion, pyrolysis, or reburn experiments. The results could be useful as well for anaerobic digestion experiments. To determine physical and thermochemical properties, composite samples of all manure, compost, and coal and lignite materials were analyzed by Hazen Research, Inc. (Golden, Colorado) for the following parameters:

- Proximate analysis: Moisture, ash, volatile solids, and fixed carbon (FC).
- Ultimate analysis: Moisture, ash, total carbon, hydrogen, nitrogen, sulfur, and oxygen (by difference).
- Higher heating value (HHV): Wet basis (w.b., as received), dry basis (d.b.), and dry ash-free (DAF) basis.
- Selected minerals: Chlorine, phosphorus, and sulfur.
- Ash elemental analysis (oxide basis): Silicon, aluminum, titanium, iron, calcium, magnesium, sodium, potassium, phosphorus, sulfur, chlorine, and carbon dioxide.
- Metals in ash ( $\text{mg kg}^{-1}$ ): Arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver.

Alkaline compounds act as catalysts in combustion and gasification, and the commercial analyses are useful for interpreting this aspect (Lindstad et al., 2004; Vipul, 2009).

For proximate, ultimate, and HHV analyses, all three

collected composite samples of each type of FB were analyzed. For analysis of metals and elemental analysis of ash, only one composite sample of the three collected samples was analyzed for each type of manure due to expense. Using the fuel analyses as input, the N, S, and ash contents were presented on an elemental and heat basis. The physical and chemical compositions of the feedlot biomass, lignite, and coal influence how effectively these potential fuels can be utilized separately or as blends with coal or lignite.

## PARTIAL COMPOSTING

Composting in windrows for a limited time period was used as an available means of stabilization, moisture reduction, and homogenization of collected FB materials. When necessary to initiate composting, the moisture content of the windrow-stored FB was raised by adding water, using a tractor-towed tank wagon equipped with a sprayer. For instance, in June 2005, approximately 11,356 L (3,000 gal) of water was sprayed on the LA-FB windrow; a week later, following rainfall, only 3,470 L (900 gal) of water were needed on the HA-FB windrow to initiate composting.

The LA-FB and HA-FB windrows were partially composted (PC) for 55 and 51 days, respectively, beginning on 9 or 13 June 2005 and being removed on 5 August 2005. Composite 2 kg samples comprised of ten subsamples were extracted from both windrows on 2 August 2005 and were submitted for analyses as LA-FB-PC and HA-FB-PC materials, respectively.

Work continued in fall and winter of 2006-2007 in which partially composted low-ash and high-ash beef cattle manure was stored and dried in a greenhouse at the USDA-ARS-CPRL research facility at Bushland, Texas. FB stored at the Texas A&M AgriLife Research-Amarillo/USDA-ARS Bushland research facility was processed to specifications (Annamalai et al., 2012) and then shipped to TEES/TAMU for conversion experiments.

## SOLID BIOFUEL GRINDING AND PULVERIZATION

Approximately 1,542 to 1,724 kg (3,400 to 3,800 lbs) of LA-FB-PC and 454 kg (1,000 lbs) of HA-FB-PC was pulled from the two windrows and ground in the hammer mill. For use in subsequent pilot plant reburn tests, some of the FB was further ground (pulverized) in a Vortec impact mill, which was capable of processing  $\sim 227$  kg (500 lbs) of material per hour, provided that the FB moisture content was  $\leq 10\%$  w.b. The Vortec impact mill was used to process manure to provide a finely ground finished product that was acceptable for reburn combustion testing, together with pulverized coal or lignite.

Wyoming PRB low-sulfur coal was donated by XCEL Energy from the Harrington Station coal-fired power plant in Amarillo, Texas. The coal was prepared for experiments being conducted at the Texas A&M University 29 kW ( $100,000 \text{ BTU h}^{-1}$ ) small-scale boiler burner, a 29.3 kW ( $100,000 \text{ BTU h}^{-1}$ ) reburn system used for NO<sub>x</sub> and Hg reduction studies, and 8.8 kW ( $30,000 \text{ BTU h}^{-1}$ ) gasification facilities. This coal was dried, pulverized with a hammer mill, and then processed with the Vortec impact mill to further reduce the particle size to specification ( $>70\%$  passing

a 74  $\mu\text{m}$  (No. 200) mesh sieve) for coal or lignite, or ~50% of FB passing a 74  $\mu\text{m}$  (No. 200) mesh sieve). The processed FB, Texas lignite, or PRB coal was then shipped to Annamalai et al. (2012) in College Station, Texas, or to a contracted pilot test facility.

### BULK DENSITY

Following the initial bulk sampling of harvested manure from the feedpens, bulk density of material in both the LA-FB and HA-FB windrows was determined. Bulk density was determined by two alternative standard methods: ASABE Standard S269.4 (*ASABE Standards*, 2011) and ASTM Standard D1895 (ASTM, 2003). ASABE Standard S269.4 was modified slightly by using a 0.028  $\text{m}^3$  (1  $\text{ft}^3$ ) container with inside dimensions of 30.5 cm  $\times$  30.5 cm  $\times$  30.5 cm, rather than a 0.057  $\text{m}^3$  (2  $\text{ft}^3$ ) specified container size, and the moisture content was not corrected to 0%. ASABE Standard S269.4 required the material to be poured from a height of 61 cm (2 ft) until the container was filled. Once the container was filled, all excess material was scraped off with a straight edge level with the top of the container to establish a 0.028  $\text{m}^3$  (1  $\text{ft}^3$ ) struck volume of material. The material was then dropped five times from a height of 15.24 cm (0.5 ft). Each time the container was dropped and the FB settled, more FB was added to the container and struck level with the top surface, and then the process was repeated. The manure was weighed after the fifth drop and addition of FB. This test was repeated three times with random samples each of the HA-FB and LA-FB. Three samples each of the LA-FB and HA-FB were taken to determine gravimetric moisture content after 24 h at 75°C in a drying oven.

ASTM Standard D1895 (ASTM, 2003) required the material to be compacted in a known volume. The material was poured from a height of 61 cm (2 ft) until the container was filled. Once the container was filled, all excess material was scraped off with a straight edge level with the top of the container, and then weighed. This test was repeated three times with random samples of the LA-FB and three times with random samples of the HA-FB. Three samples of both the LA-FB and the HA-FB were taken to determine moisture content, which was determined gravimetrically after drying for 24 h at 75°C in a drying oven.

### STATISTICAL ANALYSIS

The statistical analyses between the different types of feedlot biomass, PRB coal, and Texas lignite were achieved using an analysis of variance (ANOVA) statistical model (SPSS version 13.0). The ANOVA model assumptions were that all variances were equal and each probability distribution was normal. Feedlot biomass samples were paired based on the year collected, type of material (HA vs. LA, FB vs. coal, lignite vs. coal, etc.), and how the material was processed (raw manure vs. composting vs. storage time). Combustion data results were compared using dry basis values, except for HHV  $\text{kJ kg}^{-1}$  wet basis (w.b., as received). All values reported in tables 1, 2, and 4 having  $n = 3$  samples each were compared for each of the 27 pairings.

## RESULTS AND DISCUSSION

### 2005 FEEDLOT BIOMASS

The above protocols and preliminary results summarizing the 2005 feedlot manure/compost FB characterization results were reported by Sweeten et al. (2006). The fuel properties of raw cattle feedlot manure (FB-RM) harvested on 20 May to 8 June 2005 (paved pens) and on 1 to 10 June 2005 (unpaved pens) from the Texas A&M AgriLife Research/USDA-ARS research feedlot, Bushland, Texas, are presented in table 1. The differences between FB from the soil-surfaced pens yielding high-ash (HA) manure vs. the fly-ash paved pens yielding typically low-ash (LA) manure were readily evident.

This 2005 HA-FB-RM material had moisture content of 19.81%  $\pm$  1.24% w.b., ash content of 58.73%  $\pm$  1.65% d.b., volatile solids content of 33.77%  $\pm$  1.26% d.b., and HHV of only 6,304  $\pm$  78  $\text{kJ kg}^{-1}$  (2,710  $\pm$  34  $\text{BTU lb}^{-1}$ ) w.b. and 7,861  $\pm$  32  $\text{kJ kg}^{-1}$  (3,380  $\pm$  14  $\text{BTU lb}^{-1}$ ) d.b. The HHV on a dry ash-free (DAF) basis was 19,072  $\pm$  762  $\text{kJ kg}^{-1}$  (8,200  $\pm$  327  $\text{BTU lb}^{-1}$ ), which clearly showed the effect of ash on reducing HHV of as-collected FB.

The low-ash biomass from the paved feedlots (LA-FB-RM), although similar in moisture, had much lower ash content and higher volatile solids, fixed carbon, total carbon, and HHV than did the HA-FB-RM, as shown in table 1 and figure 1. The HHV of the LA-FB-RM was 13,407  $\pm$  342  $\text{kJ kg}^{-1}$  and 16,815  $\pm$  213  $\text{kJ kg}^{-1}$  (5,764  $\pm$  147  $\text{BTU lb}^{-1}$  and 7,229  $\pm$  92  $\text{BTU lb}^{-1}$ ) for wet (as received) and dry basis, respectively. On a DAF basis, the HHV averaged 21,072  $\pm$  30  $\text{kJ kg}^{-1}$  (9,059  $\pm$  13  $\text{BTU lb}^{-1}$ ). These values would place it near, but still below, the HHV values for the Texas lignite samples examined in this project (table 2).

In addition, using the ultimate analysis, the C-normalized empirical chemical formulas were derived (Annamalai and Puri, 2007) and are presented in table 3 for the 2005 and 2006 FB samples, Texas lignite, and PRB coal used in this project. Other important combustion or gasification related parameters can be derived from the data in tables 1, 2, and 4. Such parameters can include ratios of H/C, N/C, S/C, and O/C, which can help in interpreting heating values.

Bulk densities were determined only for the uncomposted FB characterized in table 1. Bulk densities showed major differences as a function of pen surfacing material. Specifically, bulk density of LA-FB-RM (at a moisture content of 6.40%  $\pm$  0.24% w.b.) averaged 512.06  $\pm$  4.7  $\text{kg m}^{-3}$  (31.97  $\pm$  0.29  $\text{lbs ft}^{-3}$ ) using ASABE Standard S269.4 as modified (*ASABE Standards*, 2011) and 429.51  $\pm$  0.49  $\text{kg m}^{-3}$  (26.81  $\pm$  0.03  $\text{lbs ft}^{-3}$ ) using ASTM Standard D1895 (ASTM, 2003). By contrast, HA-FB-RM (at 4.95%  $\pm$  0.02% w.b. moisture) exhibited bulk densities of 747.31  $\pm$  13.80  $\text{kg m}^{-3}$  (46.65  $\pm$  0.86  $\text{lbs ft}^{-3}$ ) with the modified ASABE standard and 650.67  $\pm$  11.38  $\text{kg m}^{-3}$  (40.61  $\pm$  0.71  $\text{lbs ft}^{-3}$ ) with the ASTM standard. Bulk density of the packed FB materials (involving five drops from 15.24 cm and refills) resulting from the modified ASABE standard exceeded that of the unpacked FB material from the ASTM method by approximately 19% and 15% for LA-FB and HA-FB, respectively.

**Table 1. Fuel properties of feedlot biomass (FB) harvested in 2005 from Texas A&M AgriLife Research/USDA-ARS research feedlot, Bushland, Texas, from soil-surfaced vs. paved feedpens.<sup>[a]</sup>**

Parameter	HA-FB-RM 2005 (10 June 2005)	LA-FB-RM 2005 (8 June 2005)	HA-FB-PC 2005 (2 Aug. 2005)	LA-FB-PC 2005 (2 Aug. 2005)	LA-FB-PC 2005 WR (7 June 2006)	LA-FB-PC 2005 GH (7 June 2006)	LA-FB-RM 2005 GH (7 June 2006)
Proximate and ultimate (%)							
Moisture (w.b.)	19.81 ±1.24	20.27 ±1.23	17.00 ±0.26	19.64 ±2.54	17.56 ±3.78	10.36 ±1.14	12.66 ±1.24
Ash	58.73 ±1.65	20.20 ±1.11	64.88 ±0.74	20.53 ±0.52	31.20 ±1.37	21.79 ±1.27	23.42 ±1.07
FC	7.50 ±0.45	15.24 ±0.27	4.05 ±0.95	14.36 ±0.28	12.78 ±0.78	13.81 ±0.19	13.93 ±0.08
VM	33.77 ±1.26	64.56 ±0.94	31.07 ±1.31	65.11 ±0.59	56.02 ±0.37	64.40 ±1.21	62.65 ±1.09
C	21.69 ±1.14	43.09 ±0.49	17.97 ±0.25	42.05 ±0.14	39.13 ±0.56	44.21 ±0.95	43.92 ±0.83
H	2.62 ±0.13	5.22 ±0.05	1.68 ±0.10	4.55 ±0.29	4.49 ±0.15	5.32 ±0.15	5.14 ±0.10
N	1.94 ±0.07	3.11 ±0.03	1.36 ±0.03	2.45 ±0.02	3.26 ±0.11	3.08 ±0.04	3.10 ±0.05
O (by diff.)	14.59 ±0.81	27.70 ±0.63	13.73 ±0.37	29.78 ±0.36	21.18 ±0.97	24.95 ±0.20	23.70 ±0.97
S	0.42 ±0.02	0.67 ±0.01	0.38 ±0.02	0.64 ±0.04	0.73 ±0.02	0.65 ±0.01	0.72 ±0.04
Cl	0.375	0.377	0.338	0.905	0.84 ±0.06	0.95 ±0.05	0.97 ±0.01
Higher heating value (HHV)							
kJ kg <sup>-1</sup> w.b.	6,304 ±78	13,407 ±342	5,207 ±114	13,267 ±446	11,804 ±868	14,865 ±368	14,293 ±380
kJ kg <sup>-1</sup> d.b.	7,861 ±32	16,815 ±213	6,274 ±141	16,507 ±39	14,306 ±424	16,582 ±248	16,364 ±213
DAF kJ kg <sup>-1</sup>	19,072 ±762	21,072 ±30	17,868 ±394	20,773 ±88	20,791 ±203	21,203 ±182	21,367 ±124
P <sub>2</sub> O <sub>5</sub> (%)							
Dry basis	1.61 ±0.04	2.59 ±0.04	1.57 ±0.01	2.73 ±0.11	4.04 ±0.17	3.17 ±0.13	3.18 ±0.19
Ash basis	2.74 ±0.08	12.87 ±0.85	2.43 ±0.05	13.30 ±0.69	12.96 ±0.83	13.90 ±0.28	13.56 ±0.18
Contaminants, energy basis							
Ash (kg GJ <sup>-1</sup> )	404.21 ±11.93	65.04 ±4.40	559.77 ±16.60	67.31 ±1.88	50.81 ±3.76	30.58 ±2.18	33.30 ±1.92
SO <sub>2</sub> (kg GJ <sup>-1</sup> )	5.84 ±0.29	4.32 ±0.12	6.50 ±0.31	4.16 ±0.26	2.39 ±0.15	1.83 ±0.01	2.06 ±0.13
Ash elemental analysis (%)							
SiO <sub>2</sub>	64.68	25.55	65.55	20.78	27.90 ±3.33	22.03 ±0.90	22.89 ±0.68
Al <sub>2</sub> O <sub>3</sub>	7.72	1.94	11.2	4.94	4.47 ±0.43	3.78 ±0.22	3.93 ±0.05
CaO	7.09	20.20	7.47	21.0	19.67 ±1.58	21.20 ±0.36	21.03 ±0.15
TiO <sub>2</sub>	0.44	0.27	0.52	0.22	0.28 ±0.03	0.24 ±0.02	0.26 ±0.02
Fe <sub>2</sub> O <sub>3</sub>	2.90	1.37	2.99	1.71	1.66 ±0.08	1.59 ±0.11	1.73 ±0.25
MgO	2.34	7.17	2.29	7.54	6.86 ±0.49	7.28 ±0.18	7.23 ±0.26
Na <sub>2</sub> O	1.38	4.94	1.38	5.26	4.25 ±0.43	4.72 ±0.15	4.23 ±0.08
K <sub>2</sub> O	4.50	12.7	4.66	14.60	12.53 ±0.84	13.57 ±0.29	13.07 ±0.32
P <sub>2</sub> O <sub>5</sub>	2.81	11.11	2.43	13.77	12.96 ±0.83	13.90 ±0.28	13.56 ±0.18
SO <sub>3</sub>	1.06	4.46	1.30	4.47	3.89 ±0.15	4.43 ±0.25	4.86 ±0.28
Cl	0.68	5.02	0.41	5.07	4.34 ±0.75	5.06 ±0.05	4.82 ±0.25
Metals in ash (mg kg <sup>-1</sup> )							
Arsenic	4.12	3.96	3.85	2.81	5.12 ±0.87	3.71 ±0.30	3.76 ±0.14
Barium	669	2,620	800	700	793 ±121	790 ±63	822 ±53.51
Cadmium	<20	2	3.8	8.2	5.97 ±0.40	5.23 ±0.40	6.10 ±0.17
Chromium	<20	20	30	40	53.3 ±5.77	63.3 ±5.77	193 ±222
Lead	20	20	27	15	15 ±5.29	16.00 ±4.58	22.67 ±5.51
Mercury	<0.01	<0.01	0.03	0.04	0.05 ±0.03	0.02 ±0.00	0.02 ±0.00
Selenium	<2	2	<2	4	5.33 ±1.53	5.67 ±1.53	4.67 ±0.58
Silver	<2	<2	<2	<2	2	2	<2

<sup>[a]</sup> FB materials represent as-collected/mixed raw manure (RM) and uncomposted material or partially composted (PC) material from two types of feedlot pen surfaces: soil-surfaced pens (designated as high ash, HA) or paved pens with crushed bottom ash/fly-ash surfaces (designated as low ash, LA). Samples taken after longer-term storage (9 to 12 months) are additionally identified as windrow-stored (WR) or greenhouse-stored (GH). All values are reported as means ±SD when *n* = 3 samples were available, and all values are reported on dry basis (d.b.) unless noted. Analyses without standard deviations (±) were equal-weight composites of the samples analyzed in the proximate and ultimate results listed in the table.

LA-FB from paved feedlots had a bulk density only two-thirds that of HA-FB from unpaved/soil-surfaced feedlots, which is attributable to the higher soil/ash content of the latter material.

Uncomposted FB displayed differences in elemental composition of sample-ash depending on type of feedlot surfacing material. Compared to HA-FB, the LA-FB appeared to contain lower Si, Al, Fe, and Ti, but was higher in Ca, Mg, Na, K P, S, Cl, and Ba (table 1). These results should be interpreted with caution, as they were based on only one composite sample per FB type.

#### PARTIAL COMPOSTING EFFECTS ON 2005 FB

The windrowed manure from the soil-surfaced pens and the crushed fly-ash surfaced pens were resampled on 2 Au-

gust 2005 following 51-day and 55-day composting periods, respectively, alongside the Texas A&M AgriLife Research/USDA-ARS research feedlot at Bushland, Texas. The analytical data are shown in table 1. Compared with HA-FB-RM, the HA-FB-PC after 51 days of composting (table 1) showed higher values of ash (64.88% ±0.74% d.b.) and lower values of moisture (17.0% ±0.26% w.b.), volatile matter (31.07% ±1.31% d.b.), fixed carbon (FC; 4.05% ±0.95%), and HHV at 5,207 ±114 kJ kg<sup>-1</sup> w.b., 6,274 ±141 kJ kg<sup>-1</sup> d.b., and 17,868 kJ kg<sup>-1</sup> DAF (2,239 ±49 BTU lb<sup>-1</sup> w.b., 2,697 ±60 BTU lb<sup>-1</sup> d.b., and 7,682 ±169 BTU lb<sup>-1</sup> DAF). These differences were statistically significant for HHV wet and dry basis, ash, FC, C, H, and N, as discussed in a subsequent section.

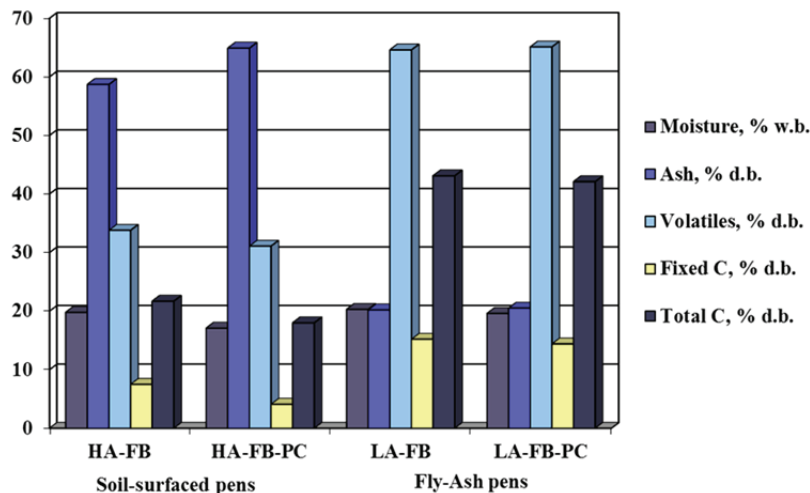


Figure 1. Spring-harvested FB in 2005 (wet year): Effects of pen surfacing and composting on moisture, ash, volatile, fixed carbon, and total carbon (Sweeten et al., 2006).

The LA-FB-RM material was resampled and analyzed after 55 days of windrow composting, at which point it was considered partially composted (LA-FB-PC). Table 1 shows essentially the same average moisture content ( $19.64\% \pm 2.54\%$  w.b.), ash content ( $20.53\% \pm 0.52\%$  d.b.), and volatile solids ( $65.11\% \pm 0.59\%$  d.b.) as before composting. Fixed C and total C were lowered slightly to  $14.36\% \pm 0.28\%$  and  $42.05\% \pm 0.14\%$  d.b., respectively. HHV was slightly less at  $13,267 \pm 446$  kJ kg<sup>-1</sup>,  $16,507 \pm 39$  kJ kg<sup>-1</sup>, and  $20,773 \pm 88$  kJ kg<sup>-1</sup> ( $5,704 \pm 192$  BTU lb<sup>-1</sup>,  $7,097 \pm 17$  BTU lb<sup>-1</sup>, and  $8,931 \pm 38$  BTU lb<sup>-1</sup>) on a wet (as received), dry, and DAF basis, respectively. These differences were significantly different for HHV (DAF), FC, C, H, N, and O.

Comparing 2005 harvested and partially composted HA-FB-PC from soil-surfaced pens versus LA-FB-PC from fly-ash surfaced pens, several large differences stand out in terms of HHV (fig. 2) as well as volatile matter and ash content (fig. 1) on both wet (as received) and dry basis. Most of the comparative differences are not surprising, given the continual loss of volatile matter that can occur during composting. For all parameters, LA-FB-PC was significantly different from HA-FB-PC, as shown subsequently. Partially composted manure from the soil-surfaced pens harvested in early June 2005, after a wet winter and spring, had almost three times the ash content and less than half the wet basis (as received) higher heating value (HHV) and volatile solids as FB similarly collected from the paved pens. The proximate and ultimate analyses showed that the HHV was reduced by 10% due to loss of volatile matter during 51 or 55 days of composting. The results did not indicate major differences in elemental composition of sample ash for either HA-FB or LA-FB resulting from partial composting, but an insufficient number of samples were analyzed for elemental composition to detect trends.

#### EFFECTS OF STORAGE ON 2005 FB

Research continued using bulk FB samples of 953 to 1,542 kg (2,100 to 3,400 lbs) from soil-surfaced and fly-ash paved feedpens that were retained from the winter-spring

2005 feeding trial and summer 2005 FB characterization study. This material was again sampled on 7 June 2006 and analyzed for the same parameters to determine effects of 10 to 12 months additional storage in a windrow or greenhouse on fuel quality parameters. The material was designated as windrow or greenhouse stored depending on the conditions of storage, i.e., outdoor or indoor.

The 2005 LA-FB-PC windrow-stored manure continued to lose volatile solids and to concentrate ash and minerals during the subsequent 12-month continuous storage in the windrow (table 1), although no further moisture addition or turning occurred. When sampled on 7 June 2006, final moisture content was  $17.56\% \pm 3.78\%$  w.b., as shown in table 1, which was slightly lower (from  $19.64\% \pm 2.54\%$ ) than a year earlier (table 1). Ash increased further to  $31.20\% \pm 1.37\%$  d.b. (from  $20.53\% \pm 0.52\%$ ), and decreases occurred in volatile solids ( $56.02\% \pm 0.37\%$ ), fixed C ( $12.78\% \pm 0.78\%$  d.b.), total C ( $39.13\% \pm 0.56\%$ ), hydrogen ( $4.49\% \pm 0.15\%$  d.b.), and chlorine ( $0.84\% \pm 0.06\%$ ) compared with year-earlier levels. Slight concentration increases appeared in nitrogen ( $3.26\% \pm 0.11\%$  d.b.) and sulfur ( $0.73\% \pm 0.02\%$ ). Heating value averaged  $11,804 \pm 868$  kJ kg<sup>-1</sup> ( $5,075 \pm 373$  BTU lb<sup>-1</sup>) w.b. (11% lower than a year earlier),  $14,306 \pm 424$  kJ kg<sup>-1</sup> d.b. ( $6,850 \pm 182$  BTU lb<sup>-1</sup>) d.b., and  $20,791 \pm 203$  kJ kg<sup>-1</sup> ( $8,939 \pm 87$  BTU lb<sup>-1</sup>) DAF basis. Ash analysis showed elemental oxide values as follows: silicon ( $27.90\% \pm 3.33\%$  d.b.), alumina ( $4.47\% \pm 0.43\%$  d.b.), calcium ( $19.67\% \pm 1.58\%$  d.b.), and sodium ( $4.25\% \pm 0.43\%$  d.b.). Potassium and phosphorus oxide values in ash residue were both high at  $12.53\% \pm 0.84\%$  d.b. and  $12.96\% \pm 0.83\%$  d.b., respectively, and sulfur oxide was present at  $3.89\% \pm 0.15\%$  d.b. Metals in ash were relatively low, except possibly for chromium.

By comparison, the 2005 LA-FB-PC greenhouse-stored manure dried substantially (table 1) but appeared to have almost ceased to gain ash or to lose volatile solids, fixed carbon, total carbon, or heating value compared with the same LA-FB-PC collected from the windrows on 2 August 2005, almost ten months earlier. As of the 7 June 2006

**Table 2. Fuel properties of Texas lignite and Wyoming Powder River basin (PRB) coal (sampling date was 10 October 2005).<sup>[a]</sup>**

Parameter	Texas Lignite	PRB Coal
Proximate and ultimate (%)		
Moisture (w.b.)	38.34 ±0.34	32.88 ±0.36
Ash	18.59 ±0.85	8.40 ±3.11
FC	41.21 ±0.80	49.15 ±2.15
VM	40.20 ±0.53	42.45 ±1.02
C	60.30 ±0.92	69.32 ±2.82
H	3.44 ±0.14	4.06 ±0.13
N	1.11 ±0.02	0.98 ±0.04
O by (diff.)	15.58 ±0.44	16.83 ±0.29
S	0.98 ±0.15	0.41 ±0.03
Cl	0.016	0.013
Higher heating value (HHV)		
kJ kg <sup>-1</sup> w.b.	14,289 ±295	18,196 ±656
kJ kg <sup>-1</sup> d.b.	23,172 ±395	27,114 ±1,058
DAF kJ kg <sup>-1</sup>	28,462 ±195	29,596 ±226
P <sub>2</sub> O <sub>5</sub> (%)		
Dry basis	0.02 ±0.00	0.05 ±0.01
Ash basis	0.13 ±0.01	0.57 ±0.14
Contaminants, energy basis		
Ash (kg GJ <sup>-1</sup> )	43.43 ±2.72	16.93 ±7.03
SO <sub>2</sub> (kg GJ <sup>-1</sup> )	4.60 ±0.02	1.64 ±0.05
Ash elemental analysis (%)		
SiO <sub>2</sub>	48.72	31.73
Al <sub>2</sub> O <sub>3</sub>	16.04	17.27
TiO <sub>2</sub>	0.85	1.35
Fe <sub>2</sub> O <sub>3</sub>	7.44	4.61
CaO	11.70	22.20
MgO	1.93	5.62
Na <sub>2</sub> O	0.29	1.43
K <sub>2</sub> O	0.61	0.67
P <sub>2</sub> O <sub>5</sub>	0.10	0.80
SO <sub>3</sub>	10.80	10.40
Cl	<0.01	<0.01
Metals in ash (mg kg <sup>-1</sup> )		
Arsenic	24.70	17.6
Barium	1,590	6,230
Cadmium	3.40	5.20
Chromium	98.00	110.00
Lead	47.00	130.00
Mercury	0.01	<0.01
Selenium	<2	<2
Silver	<2	<2

<sup>[a]</sup> Lignite samples were supplied by TXU Energy, Dallas, Texas, and PRB coal samples were supplied by XCEL Energy from the Harrington Station coal-fired power plant in Amarillo, Texas. All values are reported as means ±SD when *n* = 3 samples were available, and all values are reported on dry basis (d.b.) unless noted. Analyses without standard deviations (±) were equal-weight composites of the samples analyzed in the proximate and ultimate results.

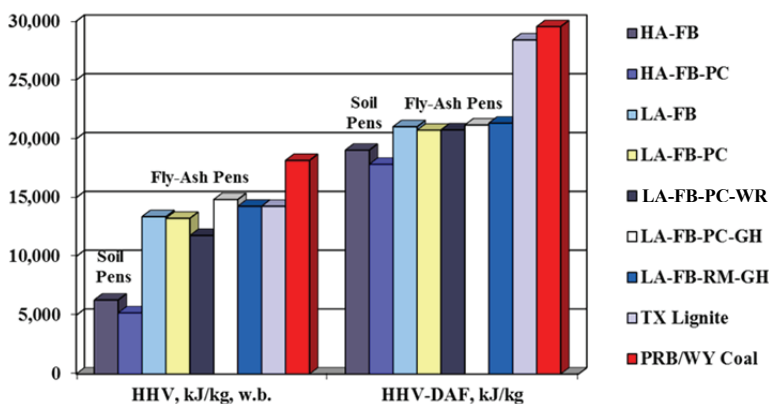
**Table 3. Empirical formulas for feedlot biomass, lignite, and coal based on ultimate and proximate values from tables 1, 2, and 4.<sup>[a]</sup>**

Material	Empirical Formula
2005 HA-FB-RM	CH <sub>1.44</sub> N <sub>0.077</sub> O <sub>0.51</sub> S <sub>0.0073</sub>
2005 LA-FB-RM	CH <sub>1.44</sub> N <sub>0.062</sub> O <sub>0.48</sub> S <sub>0.0058</sub>
2005 HA-FB-PC	CH <sub>1.11</sub> N <sub>0.065</sub> O <sub>0.57</sub> S <sub>0.0079</sub>
2005 LA-FB-PC	CH <sub>1.29</sub> N <sub>0.050</sub> O <sub>0.53</sub> S <sub>0.0057</sub>
2006 HA-FB-RM	CH <sub>1.42</sub> N <sub>0.061</sub> O <sub>0.37</sub> S <sub>0.0050</sub>
2006 LA-FB-RM	CH <sub>1.41</sub> N <sub>0.058</sub> O <sub>0.41</sub> S <sub>0.0041</sub>
2006 HA-FB-PC	CH <sub>1.31</sub> N <sub>0.068</sub> O <sub>0.31</sub> S <sub>0.0057</sub>
2006 LA-FB-PC	CH <sub>1.35</sub> N <sub>0.071</sub> O <sub>0.38</sub> S <sub>0.0056</sub>
Texas lignite (2005)	CH <sub>0.68</sub> N <sub>0.016</sub> O <sub>0.19</sub> S <sub>0.0062</sub>
PRB coal (2005)	CH <sub>0.70</sub> N <sub>0.012</sub> O <sub>0.1</sub> S <sub>0.0022</sub>

<sup>[a]</sup> Analyses were performed using ThermoLab software in the Coal and Biomass Energy Laboratory of the Department of Mechanical Engineering Texas A&M University, College Station, Texas.

sample date, moisture was much lower at only 10.36% ±1.14% w.b., ash was 21.79% ±1.27% d.b., volatile solids was 64.40% ±1.21% d.b., fixed C was 13.81% ±0.19% d.b., total C was 44.21% ±0.95% d.b., hydrogen was 5.32% ±0.15% d.b., and nitrogen was 3.08% ±0.04% d.b. Selected minerals showed dry basis results for sulfur (0.65% ±0.01% d.b.) and chlorine (0.95% ±0.05% d.b.). The heating value averaged 14,865 ±368 kJ kg<sup>-1</sup> (6,391 ±158 BTU lb<sup>-1</sup>) w.b., 16,582 ±248 kJ kg<sup>-1</sup> (7,129 ±107 BTU lb<sup>-1</sup>) d.b., and 21,203 ±182 kJ kg<sup>-1</sup> (9,116 ±78 BTU lb<sup>-1</sup>) DAF basis. These values were 26%, 16%, and 2% higher, respectively, than those for the 2005 LA-FB-PC windrow-stored material, possibly reflecting the low moisture content maintained in greenhouse storage. Ash analysis showed elemental oxide values as follows: silicon (22.03% ±0.90% d.b.), alumina (3.78% ±0.22% d.b.), calcium (21.20% ±0.36% d.b.), and sodium (4.72% ±0.15% d.b.). Potassium and phosphorus oxide values in ash residue were both high at 13.57% ±0.29% d.b. and 13.90% ±0.28% d.b., respectively, while sulfur oxide was present at 4.43% ±0.35% d.b. and chlorine oxide at 5.06% ±0.05% d.b. Metals in ash were relatively low, except possibly for chromium.

Uncomposted 2005 LA-FB-RM manure that had been placed in the greenhouse shortly after collection (2005 LA-FB-RM greenhouse-stored) appeared to have similar or slightly improved HHV fuel properties than the 2005 LA-FB-PC windrow-stored manure of the same origin. Selected results from the 7 June 2006 sampling (table 1) included



**Figure 2. FB harvested in 2005 (wet winter and spring): Effect of feedlot surfacing, composting, and storage on higher heating value (HHV, kJ kg<sup>-1</sup>) on wet basis (w.b., as received) and dry ash-free (DAF) basis (multiply HHV in kJ kg<sup>-1</sup> by 0.430 to obtain HHV in BTU lb<sup>-1</sup>).**

the following values: moisture (12.66%  $\pm$ 1.24% w.b.), ash (23.42%  $\pm$ 1.07% d.b.), volatile solids (62.65%  $\pm$ 1.09% d.b.), fixed C (13.93%  $\pm$ 0.08% d.b.), total carbon (43.92%  $\pm$ 0.83% d.b.), hydrogen (5.14%  $\pm$ 0.10% d.b.), nitrogen (3.10%  $\pm$ 0.05% d.b.), sulfur (0.72%  $\pm$ 0.04% d.b.), and Cl (0.97%  $\pm$ 0.01%). Heating value averaged 14,293  $\pm$ 380 kJ kg<sup>-1</sup> (6,145  $\pm$ 163 BTU lb<sup>-1</sup>) w.b., 16,364  $\pm$ 213 kJ kg<sup>-1</sup> (7,035  $\pm$ 92 BTU lb<sup>-1</sup>) d.b., and 21,367  $\pm$ 124 kJ kg<sup>-1</sup> (9,186  $\pm$ 53 BTU lb<sup>-1</sup>) DAF basis. These HHV values were only 4%, 1%, and 0% lower, respectively, than those for the corresponding PC greenhouse-stored material and were 22%, 14%, and 3% higher, respectively, than those for the wind-row-stored manure. Ash analysis showed elemental oxide values as follows: silicon (22.89%  $\pm$ 0.68% d.b.), alumina (3.93%  $\pm$ 0.05% d.b.), calcium (21.03%  $\pm$ 0.15% d.b.), and sodium (4.23%  $\pm$ 0.08% d.b.). Potassium and phosphorus oxide values in ash residue were both high at 13.07%  $\pm$ 0.32% d.b. and 13.56%  $\pm$ 0.18% d.b., respectively, while sulfur oxide was present at 4.86%  $\pm$ 0.28% d.b. and chlorine oxide at 4.82%  $\pm$ 0.25% d.b. These ash analysis values were almost identical to those for the 2005 LA-FB-PC greenhouse-stored material. Metals in ash were relatively low, except possibly for chromium, which was higher at 193  $\pm$ 222 mg kg<sup>-1</sup>. These data appeared to show that greenhouse storage of raw manure at 12.66%  $\pm$ 0.24% w.b. moisture content can substitute for partial composting and/or wind-row storage as a method of quickly stabilizing and preserving most biofuel-related properties, which is an important finding.

## 2006 FEEDLOT BIOMASS

A third phase of the study involved harvesting and characterizing 2006 feedlot manure. In a similar fashion, we harvested, processed, sampled, and characterized manure (FB) from a concluded cattle feeding trial at the Texas A&M AgriLife Research/USDA-ARS research feedlot at Bushland, Texas, for use as fuel source for a planned pilot plant reburn combustion study in fall 2006 by Annamalai et al. (2012). These FB data represented a second (2006) replicate of the 2005 study and were designed to enhance the databases on HA-FB and LA-FB as raw, partially composted, or stored biomass. This 2006 manure was harvested/collected in late May and early June 2006, following the winter 2005 to spring 2006 cattle feeding trials, which were conducted during one of the driest nine-month periods ever recorded, 10.67 cm (4.2 in.) of precipitation, based on official Amarillo weather records dating back to the late 1880s. Hence, there was very limited opportunity for soil entrainment or runoff, except where water spillage might have occurred around drinking water troughs. The 2006 manure was harvested and FB samples were collected, processed, and analyzed using the same protocols used for the 2005 FB. Major differences were evident in the 2006 harvested manure compared with the 2005 harvested manure, and these differences could be largely attributed to the much higher precipitation in 2004–2005 vs. much lower precipitation in the 2005–2006 cattle feeding period preceding the FB harvest.

The 2006 HA-FB-RM were composite samples taken from the feedlot biomass (FB) harvested on 7 June 2006 from soil-surfaced feedpens at the Texas A&M AgriLife Research/USDA-ARS research feedlot at Bushland, Texas, following cattle feeding trials that ended in May 2006. Samples were taken of this uncomposted raw manure immediately after removal from feedpens and prior to composting and submitted for analysis. These data are summarized in table 4 and figures 3 and 4. Selected results included the following values: moisture (27.31%  $\pm$ 0.61% w.b.), ash (45.23%  $\pm$ 3.09% d.b.), volatile solids (44.71%  $\pm$ 2.55% d.b.), fixed C (10.06%  $\pm$ 0.59% d.b.), total C (32.34%  $\pm$ 2.72% d.b.), hydrogen (3.85%  $\pm$ 0.33% d.b.), nitrogen (2.31%  $\pm$ 0.17% d.b.), sulfur (0.43%  $\pm$ 0.02% d.b.), and Cl (0.24%  $\pm$ 0.18%). Heating value (fig. 4) averaged 8,190  $\pm$ 488 kJ kg<sup>-1</sup> (3,521  $\pm$ 210 BTU lb<sup>-1</sup>) w.b., 11,267  $\pm$ 685 kJ kg<sup>-1</sup> (4,844  $\pm$ 295 BTU lb<sup>-1</sup>) d.b., and 20,566  $\pm$ 115 kJ kg<sup>-1</sup> (8,842  $\pm$ 50 BTU lb<sup>-1</sup>) DAF basis. Ash elemental analysis showed oxide-basis values as follows: silicon (quite high at 60.94%  $\pm$ 1.62% d.b.), alumina (7.68%  $\pm$ 0.39% d.b.), calcium (9.91%  $\pm$ 0.76% d.b.), magnesium (2.91%  $\pm$ 0.27% d.b.), and sodium (1.78%  $\pm$ 0.10% d.b.). Potassium oxide in ash residue was 5.94%  $\pm$ 0.48% d.b., and phosphorus oxide was 4.39%  $\pm$ 0.36% d.b., while sulfur oxide was present at 1.19%  $\pm$ 0.12% d.b. and chlorine oxide at 1.12%  $\pm$ 0.16% d.b. Metals in ash were relatively low, except possibly for chromium, which was 100  $\pm$ 44 mg kg<sup>-1</sup>. Bulk samples of the 2006 HA-FB-RM were placed in a greenhouse immediately after collection to dry to  $\leq$ 10% moisture content (w.b.) for later processing as needed for biofuel feedstock experiments.

The 2006 LA-FB-RM from the fly-ash surfaced feedpens was collected on 7 June 2006, and bulk samples were immediately placed in a greenhouse to dry to  $\leq$ 10% w.b. for processing as needed for later biofuel experiments. Samples were taken of this raw manure immediately after removal from feedpens and prior to composting. These data are included in table 4 and figures 3 and 4. Selected results from the 7 June 2006 sampling included the following values: moisture (29.25%  $\pm$ 1.12% w.b.), ash (13.58%  $\pm$ 0.85% d.b.), volatile solids (68.06%  $\pm$ 0.67% d.b.), fixed C (18.36%  $\pm$ 0.20% d.b.), total C (49.63  $\pm$ 0.41% d.b.), hydrogen (5.90%  $\pm$ 0.08% d.b.), nitrogen (3.35%  $\pm$ 0.07% d.b.), sulfur (0.54%  $\pm$ 0.07% d.b.), and Cl (0.68%  $\pm$ 0.04% d.b.). Heating value on a wet (as received) basis averaged 13,224  $\pm$ 338 kJ kg<sup>-1</sup> (5,685  $\pm$ 145 BTU lb<sup>-1</sup>) w.b. (fig. 4), influenced by the higher moisture content on the date of sampling compared to the 2005 FB material. However, the 2006 FB was very high in dry basis HHV due to low ash content, having excellent heating values of 18,690  $\pm$ 207 kJ kg<sup>-1</sup> (8,035  $\pm$ 89 BTU lb<sup>-1</sup>) d.b. and 21,627  $\pm$ 112 kJ kg<sup>-1</sup> (9,298  $\pm$ 48 BTU lb<sup>-1</sup>) DAF basis. These values very likely reflected low antecedent rainfall, with minimal decomposition on the feedlot surface prior to harvesting and absence of ash entrainment or runoff. Ash elemental analysis showed oxide-basis values as follows: silicon (unusually low at 11.51%  $\pm$ 2.33% d.b.), alumina (also low at 1.78%  $\pm$ 0.30% d.b.), calcium (20.90%  $\pm$ 0.35% d.b.), magnesium (7.97%



**Table 4. Fuel properties of feedlot biomass (FB) harvested in 2006 from Texas A&M AgriLife Research/USDA-ARS research feedlot, Bushland, Texas, from soil-surfaced vs. paved feedpens.<sup>[a]</sup>**

Parameter	HA-FB-RM 2006 (7 June 2006)	LA-FB-RM 2006 (7 June 2006)	HA-FB-PC 2006 (6 Sept. 2006)	LA-FB-PC 2006 (6 Sept. 2006)	HA-FB-RM 2006 GH (19 June 2007)	LA-FB RM 2006 GH (19 June 2007)	HA-FB-PC 2006 GH (19 June 2007)	LA-FB-PC 2006 GH (19 June 2007)
Proximate and ultimate (%)								
Moisture (w.b.)	27.31 ±0.61	29.25 ±1.12	30.55 ±1.89	43.09 ±8.88	7.69 ±0.30	12.65 ±0.41	9.53 ±0.79	13.42 ±0.03
Ash	45.23 ±3.09	13.58 ±0.85	44.32 ±4.02	21.70 ±0.69	43.15 ±3.04	13.54 ±1.56	45.07 ±1.08	20.70 ±0.20
FC	10.06 ±0.59	18.36 ±0.20	11.01 ±1.06	17.74 ±0.12	11.09 ±1.26	18.91 ±1.17	10.71 ±0.73	17.18 ±1.20
VM	44.71 ±2.05	68.06 ±0.67	44.67 ±2.96	60.56 ±0.60	45.76 ±2.08	67.55 ±1.06	44.21 ±0.35	62.12 ±1.22
C	32.34 ±2.72	49.63 ±0.41	34.20 ±2.66	45.70 ±0.27	30.77 ±1.96	47.46 ±0.32	30.08 ±0.81	44.49 ±0.37
H	3.85 ±0.33	5.90 ±0.08	3.77 ±0.23	5.20 ±0.16	3.43 ±0.25	4.96 ±0.20	3.01 ±0.18	4.66 ±0.04
N	2.31 ±0.17	3.35 ±0.07	2.72 ±0.10	3.76 ±0.21	2.55 ±0.15	3.78 ±0.09	2.94 ±0.02	3.84 ±0.04
O (by diff.)	15.84 ±0.68	27.00 ±0.66	14.46 ±3.56	22.96 ±0.59	19.73 ±0.93	29.59 ±1.17	18.33 ±0.71	25.72 ±0.16
S	0.43 ±0.02	0.54 ±0.07	0.52 ±0.02	0.68 ±0.06	0.38 ±0.01	0.67 ±0.14	0.57 ±0.04	0.59 ±0.13
Cl	0.24 ±0.18	0.68 ±0.04	0.40 ±0.01	0.70 ±0.08	-	-	-	0.82 ±0.01
Higher heating value (HHV)								
kJ kg <sup>-1</sup> w.b.	8,190 ±488	13,224 ±338	7,755 ±850	9,454 ±1,560	10,410 ±861	16,049 ±427	10,021 ±107	14,153 ±70
kJ kg <sup>-1</sup> d.b.	11,267 ±685	18,690 ±207	11,154 ±1,024	16,595 ±263	11,280 ±966	18,372 ±451	11,077 ±92	16,347 ±83
DAF kJ kg <sup>-1</sup>	20,566 ±115	21,627 ±112	20,014 ±398	21,195 ±184	19,817 ±654	21,248 ±172	20,170 ±242	20,613 ±63
P <sub>2</sub> O <sub>5</sub> (%)								
Dry basis	1.99 ±0.25	2.02 ±0.10	2.06 ±0.09	2.30 ±0.14	-	-	-	2.20 ±0.06
Ash basis	4.39 ±0.36	14.92 ±0.51	4.67 ±0.25	10.61 ±0.88	-	-	-	10.63 ±0.21
Contaminants, energy basis								
Ash (kg GJ <sup>-1</sup> )	218.34 ±29.03	39.33 ±2.85	217.29 ±38.08	70.80 ±3.28	208.86 ±33.48	39.96 ±5.58	220.18 ±7.08	68.51 ±0.97
SO <sub>2</sub> (kg GJ <sup>-1</sup> )	4.07 ±0.22	3.11 ±0.42	5.09 ±0.30	4.43 ±0.42	3.64 ±0.21	3.91 ±0.82	5.580 ±0.47	3.90 ±0.88
Ash elemental analysis (%)								
SiO <sub>2</sub>	60.94 ±1.62	11.51 ±2.33	59.09 ±0.12	29.15 ±3.93	-	-	-	-
Al <sub>2</sub> O <sub>3</sub>	7.68 ±0.39	1.78 ±0.30	8.52 ±0.34	5.25 ±0.73	-	-	-	-
TiO <sub>2</sub>	0.35 ±0.02	0.11 ±0.03	0.45 ±0.00	0.31 ±0.10	-	-	-	-
Fe <sub>2</sub> O <sub>3</sub>	2.29 ±0.06	1.06 ±0.13	2.59 ±0.07	1.97 ±0.25	-	-	-	-
CaO	9.91 ±0.76	20.90 ±0.35	9.86 ±0.46	18.50 ±2.12	-	-	-	-
MgO	2.91 ±0.27	7.97 ±0.06	3.08 ±0.14	6.88 ±0.94	-	-	-	-
Na <sub>2</sub> O	1.78 ±0.10	5.57 ±0.13	1.72 ±0.07	4.42 ±0.68	-	-	-	-
K <sub>2</sub> O	5.94 ±0.48	20.27 ±0.57	5.85 ±0.22	14.10 ±1.47	-	-	-	-
P <sub>2</sub> O <sub>5</sub>	4.39 ±0.36	14.92 ±0.51	4.67 ±0.25	10.61 ±0.88	-	-	-	-
SO <sub>3</sub>	1.19 ±0.12	5.53 ±0.52	1.67 ±0.14	4.20 ±0.42	-	-	-	-
Cl	1.12 ±0.16	6.58 ±0.32	0.89 ±0.03	4.07 ±0.51	-	-	-	-
Metals in ash (mg kg <sup>-1</sup> )								
Arsenic	3.62 ±0.22	2.50 ±0.82	8.25 ±6.71	4.01 ±1.06	-	-	-	-
Barium	615 ±61	326 ±83	670 ±52	704 ±347	-	-	-	-
Cadmium	4.77 ±0.45	5.30 ±0.26	1.00 ±0.00	1.00 ±1.00	-	-	-	-
Chromium	100.0 ±43.59	166.67 ±40.41	20.0 ±0.00	26.67 ±5.77	-	-	-	-
Lead	14.33 ±3.21	10.67 ±1.15	21.00 ±3.00	24.67 ±4.51	-	-	-	-
Mercury	0.03 ±0.00	0.03 ±0.00	<0.01	0.03 ±0.02	-	-	-	-
Selenium	<2	3.00 ±0.00	<2	2.50 ±0.71	-	-	-	-
Silver	<2	2.00 ±0.00	2.00 ±0.00	<2	-	-	-	-

<sup>[a]</sup> FB materials represent as-collected/mixed raw manure (RM) and uncomposted material or partially composted (PC) material from two types of feedlot pen surfaces: soil-surfaced pens (designated as high ash, HA) or paved pens with crushed bottom ash/fly-ash surfaces (designated as low ash, LA). Samples taken after longer-term storage (9 to 12 months) were additionally identified as greenhouse-stored (GH). All values are reported as means ±SD when *n* = 3 samples were available, and all values are reported on dry basis (d.b.) unless noted. Analyses without standard deviations (±) were equal-weight composites of the samples analyzed in the proximate and ultimate results. Blank cells indicate analyses not available.

±0.06% d.b.), and sodium (5.57% ±0.13% d.b.). Potassium oxide in ash residue was very high at 20.27% ±0.57% d.b., and phosphorus oxide was also high at 14.92% ±0.51% d.b., while sulfur oxide was present at 5.53% ±0.52% d.b. and chlorine oxide at 6.58% ±0.32% d.b. Metals in ash were relatively low, except possibly for chromium, which was higher than most prior samples at 167 ±40 mg kg<sup>-1</sup>.

#### PARTIAL COMPOSTING EFFECTS ON 2006 FB

After 91 days of windrow partial composting, which ended on 6 September 2006, the HA-FB-PC for 2006 reflected similar properties as the HA-FB-RM for 2006 from which it was derived (table 4). The moisture content was slightly higher (to 30.55% ±1.89% w.b.), which may have contribut-

ed to a 5% reduction in HHV to 7,755 ±850 kJ kg<sup>-1</sup> (3,334 ±365 BTU lb<sup>-1</sup>) w.b., while both dry and DAF basis HHV were reduced very slightly (1% and 3%, respectively). Ash elemental analyses were very similar with the exception of chromium, which showed to be less in the HA-FB-PC for 2006 for unknown reasons.

By contrast, the 2006 LA-FB-PC (table 4), likewise resampled on 6 September 2006, showed much higher moisture (43.09% ±8.88% w.b.) compared with the 7 June 2006 LA-FB-RM (29.25% ±1.12% w.b.), with a higher than expected standard deviation. The higher moisture content may have led to a higher rate of composting than for the corresponding 2006 HA-FB-PC material and hence greater than expected loss of total carbon and volatile mat-

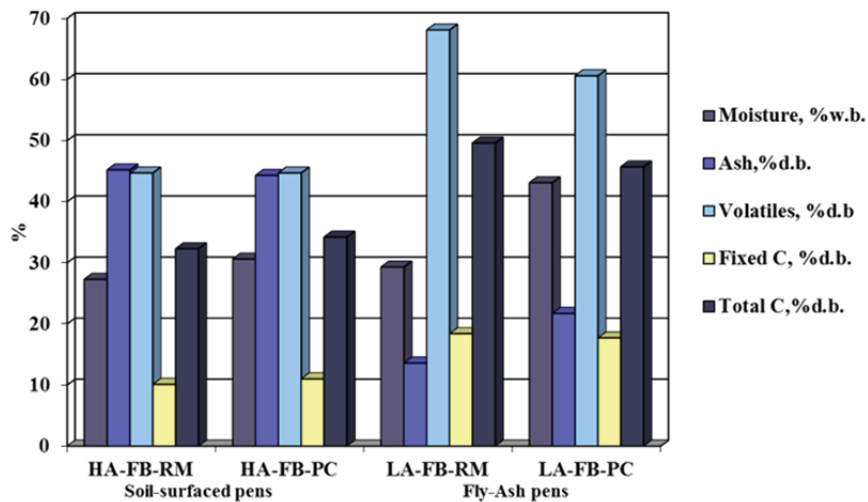


Figure 3. Spring-harvested FB for 2006 (dry year): Effects of pen surfacing and composting on moisture, ash, volatile matter, fixed carbon, and total carbon.

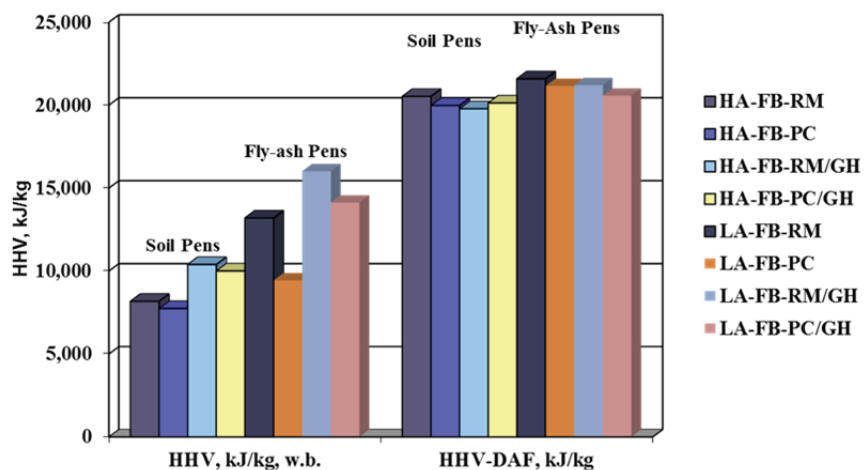


Figure 4. FB harvested in 2006 (dry winter and spring): Effect of feedlot surfacing, composting, or storage on higher heating value (HHV, kJ kg<sup>-1</sup>) wet basis (w.b., as received) and dry ash-free (DAF) basis.

ter along with a substantial increase in ash to 21.07%  $\pm$  0.69% d.b., compared with 13.58%  $\pm$  0.85% d.b. for the 2006 LA-FB-RM. Both the higher moisture and ash together with carbon loss produced a 29% reduction in HHV to 9,454  $\pm$  1,560 kJ kg<sup>-1</sup> (4,064  $\pm$  671 BTU lb<sup>-1</sup>) w.b., 16,595  $\pm$  263 kJ kg<sup>-1</sup> (7,135  $\pm$  113 BTU lb<sup>-1</sup>) d.b., and 21,195  $\pm$  184 kJ kg<sup>-1</sup> (9,112  $\pm$  79 BTU lb<sup>-1</sup>) DAF basis. However, the ash elemental analyses showed a more than two-fold increase in both silicon (SiO<sub>2</sub>) and alumina (Al<sub>2</sub>O<sub>3</sub>) as percent dry basis (d.b.), along with 24%, 29%, and 39% d.b. reductions for sulfur (SO<sub>3</sub>), potassium (K<sub>2</sub>O), and phosphorus (P<sub>2</sub>O<sub>5</sub>). These higher mineral contents may have been related to the greater ash content of the 2006 LA-FB-PC and may reflect uncertainties of sampling and resampling from bulk windrows in the field.

#### EFFECTS OF STORAGE ON 2006 FB

On 15 May to 19 June 2007, more than a year after initial harvest of the HA-FB-RM and LA-FB-RM from the research feedlot and 8 to 9 months after partial composting was ceased, material was resampled from the on-site

greenhouse storage. As shown in table 4, the 2006 HA-FB-RM greenhouse-stored material had dried to <10% w.b. and retained slightly lower ash and slightly higher volatile matter than the 2006 HA-FB-PC material, which had been likewise greenhouse-stored since 6 September 2006. The wet-basis HHV had increased substantially by 27% to 29%, apparently due to greenhouse drying, and the 12-month-old feedlot biomass had attained final HHV values of 10,410  $\pm$  861 kJ kg<sup>-1</sup> (4,476  $\pm$  370 BTU lb<sup>-1</sup>) w.b., and 10,021  $\pm$  107 kJ kg<sup>-1</sup> (4,308  $\pm$  46 BTU lb<sup>-1</sup>) w.b. for the 2006 HA-FB-RM and HA-FB-PC greenhouse-stored materials, respectively.

The counterpart low-ash material from the paved feedpens likewise showed major improvement in HHV values, which is attributed to the moisture reduction to 12.65%  $\pm$  0.41% and 13.42%  $\pm$  0.03% w.b. for the 2006 LA-FB-RM and LA-FB-PC greenhouse-stored materials, respectively, as shown in table 4. The final HHV values were: 16,049  $\pm$  427 kJ kg<sup>-1</sup> (6,900  $\pm$  183 BTU lb<sup>-1</sup>) w.b. and 14,153  $\pm$  70 kJ kg<sup>-1</sup> (6,085  $\pm$  30 BTU lb<sup>-1</sup>) w.b., respectively. Following 12 months of greenhouse storage, dry basis HHV

values were  $18,372 \pm 451 \text{ kJ kg}^{-1}$  ( $7,899 \pm 194 \text{ BTU lb}^{-1}$ ) d.b. and  $16,347 \pm 83 \text{ kJ kg}^{-1}$  ( $7,028 \pm 36 \text{ BTU lb}^{-1}$ ) d.b., respectively. The DAF-basis HHV values also decreased slightly to  $21,248 \pm 172 \text{ kJ kg}^{-1}$  ( $9,135 \pm 74 \text{ BTU lb}^{-1}$ ) and  $20,613 \pm 63 \text{ kJ kg}^{-1}$  ( $8,862 \pm 27 \text{ BTU lb}^{-1}$ ) for the 2006 LA-FB-RM and LA-FB-PC greenhouse-stored materials, respectively. Ash elemental and metal content analyses were not available for the 15 May or 19 June 2007 resampling dates.

### COMBUSTION-RELATED DERIVED PROPERTIES OF FB MATERIALS

An analysis was conducted to determine the maximum allowable ash content in a biofuel so that the biofuel could be used for a desired application. A particular focus was placed on the higher heating value (HHV). In a dynamic biofuel feedstock market, incremental HHV values represent an incremental return on investment (ROI) from paved pens. Designing or managing cattle feedlots for improved biofuel properties of FB could become economically justifiable in specific cases, depending on the availability and differential costs of FB compared with fossil fuels, together with the potential for utilizing or marketing the produced bioenergy. This research addressed biofuel properties that are important to determining the technical feasibility of such projects. Specifically, the 2005 and 2006 LA-FB-RM and LA-FB-PC data represent relatively high HHV values that may be attainable from commercial open beef cattle feedlots, without implying that feedlots should be paved to provide high-quality FB biofuel feedstock. The HHV values we obtained for LA-FB with or without composting or storage might be approximated by uniform manure harvesting in relatively dry seasons from conventional unpaved soil pens using precision equipment, trained operators, maintaining dry storage, and/or further processing, such as post-harvest ash separation. Managing feedyard surfaces for higher HHV may require extra fuel or labor, but it also

represents a potential ROI.

From previous experiments (Sweeten et al., 2003), FB samples pointed to a relatively narrow range of HHV values on a DAF basis approaching  $19,730$  to  $19,962 \text{ kJ kg}^{-1}$  ( $8,500$  to  $8,600 \text{ BTU lb}^{-1}$ ) for typical FB collected from an open soil-surfaced cattle feedlot. A linear relationship has been assumed for conservatively estimating HHV on a wet basis (w.b., as received) as a function of moisture and ash contents as follows:

$$\text{HHV (w.b.)} = \text{HHV-DAF} \times \frac{[(100 - \text{moisture \% w.b.}) \times (100 - \text{ash \% d.b.})]}{10,000}$$

According to this relationship, the heating value is reduced directly as ash and moisture are added through natural or extraneous circumstances. Since heat must be supplied to evaporate moisture, using thermochemistry we can estimate the minimum heating value as  $6,280 \text{ kJ kg}^{-1}$  ( $2,700 \text{ BTU lb}^{-1}$ ) so that the fluidized bed conversion unit could be maintained between  $1,000 \text{ K}$  to  $1,200 \text{ K}$  ( $1,340^\circ\text{F}$  to  $1,700^\circ\text{F}$ ) (Annama-lai and Puri, 2007).

Such a minimum heating value can be obtained for various ash and moisture contents, as shown in figure 5. This graph was based on the premise that the DAF heating value of animal waste based biomass fuels remains relatively constant at about  $19,800$  to  $20,000 \text{ kJ kg}^{-1}$  ( $8,500$  to  $8,600 \text{ BTU lb}^{-1}$ ), as determined previously (Sweeten et al., 2003).

In the present study, our findings showed that HHV-DAF of FB ranged from  $17,000$  to  $22,000 \text{ kJ kg}^{-1}$  ( $7,296$  to  $9,442 \text{ BTU lb}^{-1}$ ) with an average of  $20,500 \text{ kJ kg}^{-1}$  ( $8,798 \text{ BTU lb}^{-1}$ ). The HHV-DAF of FB was found to be somewhat variable, as shown in the histograms in figure 6. The HHV-DAF of low-ash FB fell into a narrower range than that of high-ash FB. The modes of the HHV-DAF dis-

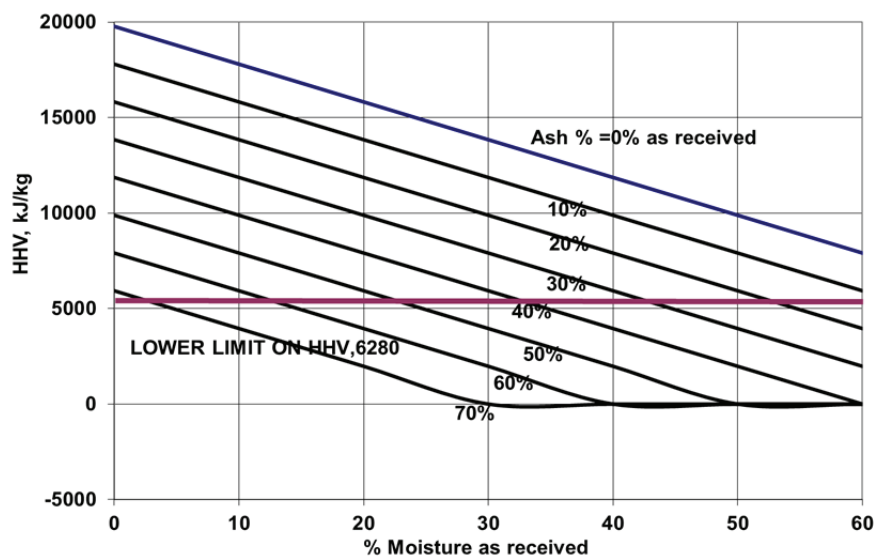


Figure 5. Allowable moisture and ash contents to achieve a heating value of  $6,280 \text{ kJ kg}^{-1}$  ( $2,700 \text{ BTU lb}^{-1}$ ) for feedlot biomass fuels, assuming a median value of  $20,000 \text{ kJ kg}^{-1}$  ( $8,600 \text{ BTU lb}^{-1}$ ) dry ash-free basis.

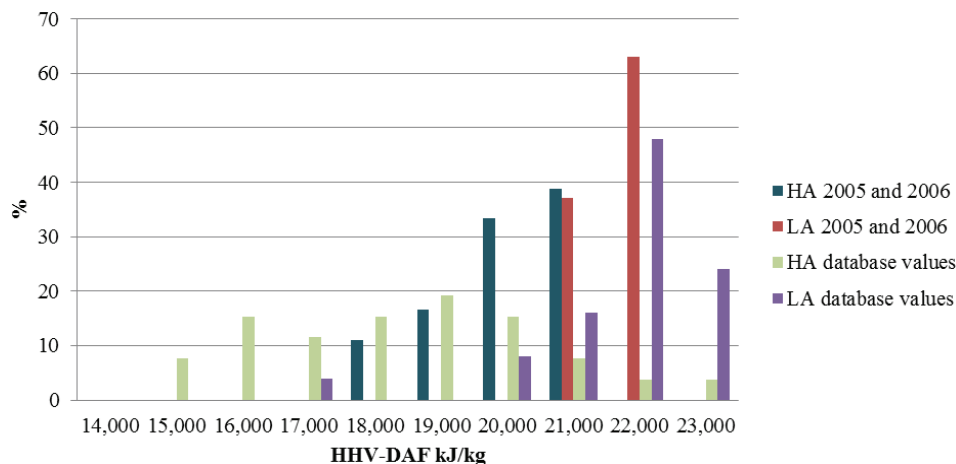


Figure 6. Distribution of higher heating values (HHV,  $\text{kJ kg}^{-1}$ ) on a dry ash-free (DAF) basis for low-ash (LA) and high-ash (HA) cattle feedlot biomass 2005 and 2006 as compared to unpublished values from the Texas A&M AgriLife Research (Amarillo) combustion database.

tributions of high-ash and low-ash FB were  $20,500 \text{ kJ kg}^{-1}$  ( $8,798 \text{ BTU lb}^{-1}$ ) and  $21,500 \text{ kJ kg}^{-1}$  ( $9,227 \text{ BTU lb}^{-1}$ ), respectively. All of the low-ash FB values were between  $20,000$  to  $22,000 \text{ kJ kg}^{-1}$  ( $8,584$  to  $9,442 \text{ BTU lb}^{-1}$ ) DAF, while 72% of the high-ash FB values were between  $19,000$  to  $21,000 \text{ kJ kg}^{-1}$  ( $8,155$  to  $9,013 \text{ BTU lb}^{-1}$ ) DAF, as shown in figure 6.

#### COMPARATIVE ANALYSES OF LIGNITE AND COAL VS. FEEDLOT BIOMASS MATERIALS

Texas lignite (supplied by TXU Energy) and PRB coal (supplied by XCEL Energy) were ground in a hammer mill, and proximate and ultimate analyses were determined for three composite samples of each. For comparison, samples of Texas lignite (TXL) and PRB coal were analyzed in the same manner as the FB materials. These results are shown in table 2. The major differences between TXL and PRB coal related primarily to higher ash content of the TXL. These data were used for direct comparisons of fuel values and chemical differences between TXL and PRB coal versus feedlot manure (tables 1 and 4).

As shown in table 2, moisture contents were  $38.34\% \pm 0.34\% \text{ w.b.}$  and  $32.88\% \pm 0.36\% \text{ w.b.}$  for TXL and PRB coal, respectively, which is considerably higher than for the FB materials (tables 1 and 4). Ash contents were much lower for the PRB coal:  $8.40\% \pm 3.11\% \text{ d.b.}$  vs.  $18.59\% \pm 0.85\% \text{ d.b.}$  for TXL. The latter value is similar to or only slightly lower than that for LA-FB and LA-FB-PC. Sulfur was higher ( $0.98\% \pm 0.15\% \text{ d.b.}$ ) for TXL than for PRB coal ( $0.41\% \pm 0.03\% \text{ d.b.}$ ) or either of the FB sources. On a dry matter basis, total carbon was much higher for TXL and PRB coal ( $60.30\% \pm 0.92\%$  and  $69.32\% \pm 2.82\% \text{ d.b.}$ , respectively) than for either LA-FB or HA-FB. Nitrogen was slightly lower and P and Cl were much lower for either TXL or PRB coal compared to LA-FB or HA-FB. As expected, compared to feedlot biomass, HHV was considerably higher for both TXL and PRB coal:  $14,289 \pm 295 \text{ kJ kg}^{-1}$  and  $18,196 \pm 656 \text{ kJ kg}^{-1}$  ( $6,143 \pm 127 \text{ BTU lb}^{-1}$  and  $7,823 \pm 282 \text{ BTU lb}^{-1}$ ) wet basis (w.b.),  $23,172 \pm 395 \text{ kJ kg}^{-1}$  and  $27,114 \pm 1,058 \text{ kJ kg}^{-1}$  ( $9,962 \pm 170 \text{ BTU lb}^{-1}$  and  $11,657 \pm 455 \text{ BTU lb}^{-1}$ ) dry basis (d.b.), and  $28,462 \pm 195 \text{ kJ kg}^{-1}$

and  $29,596 \pm 226 \text{ kJ kg}^{-1}$  ( $12,236 \pm 84 \text{ BTU lb}^{-1}$  and  $12,724 \pm 97 \text{ BTU lb}^{-1}$ ) dry ash-free (DAF) basis. Elemental ash analyses appeared similar for TXL and PRB coal, but with differences in comparison to FB for several parameters (tables 1, 2, and 4). More intensive sampling and analysis are needed to establish trends.

#### STATISTICAL SUMMARY

As shown in table 5, comparing 2005 vs. 2006 raw manure with partially composted manure, there were statistically significant differences ( $p \leq 0.028$ ) between the two years for HA-FB-RM and HA-FB-PC for the following parameters: HHV (wet, dry, and DAF); ash, VM, FC, C, H, and N; and for S with respect to the HA-FB-PC material only. Wet weather in 2005 leading to muddy unpaved feed pens was believed to be the major factor causing these differences. The differences for DAF-basis HHV are not as easily explained by weather influences alone. For LA-FB-RM, the 2005 vs. 2006 comparison was not statistically significant for wet-basis HHV, but it was for the other parameters ( $p < 0.03$ ). For 2005 vs. 2006 LA-FB-PC, statistically significant values were found for both wet- and DAF-basis HHV ( $p \leq 0.023$ ) and for VM, FC, C, H, N, and O at  $p \leq 0.026$ . However, mean values showed no difference for dry-basis HHV, ash, or sulfur.

Comparing raw vs. partially composted high-ash and low-ash manure for both years, statistically significant differences were found in 2005 HA-FB for wet- and dry-basis HHV (but not for DAF) and for ash, FC, C, H, and N; in 2006 HA-FB for S and N only; in 2005 LA-FB for DAF-basis HHV, FC, C, H, N, and O; and in 2006 LA-FB for all parameters except sulfur.

Statistical comparisons of high-ash vs. low-ash manure within years by raw manure and/or partially composted FB showed that HA-FB-RM was significantly different from LA-FB-RM for both 2005 and 2006 manure for all parameters except sulfur in 2006. Similarly, comparing high-ash vs. low-ash manure that was partially composted showed that both 2005 and 2006 HA-FB-PC were significantly different from LA-FB-PC for all parameters except wet-basis HHV in 2006 manure.

**Table 5. Analysis of variance (ANOVA) results, p-values ( $\alpha = 0.05$ ).**

Comparison	HHV (kJ kg <sup>-1</sup> )										O, by diff. (% d.b.)
	Wet basis	Dry basis	DAF basis	Ash (% d.b.)	VM (% d.b.)	FC (% d.b.)	S (% d.b.)	C (% d.b.)	H (% d.b.)	N (% d.b.)	
HA-FB-RM 2005 vs. 2006	0.003	0.001	0.028	0.003	0.003	0.004	<b>0.854</b>	0.003	0.004	0.024	<b>0.109</b>
HA-FB-PC 2005 vs. 2006	0.007	0.001	0.003	0.001	0.002	0.001	0.001	0.000	0.000	0.000	<b>0.741</b>
LA-FB-RM 2005 vs. 2006	<b>0.546</b>	0.000	0.001	0.001	0.006	0.000	0.030	0.000	0.000	0.005	<b>0.252</b>
LA-FB-PC 2005 vs. 2006	0.015	<b>0.595</b>	0.023	<b>0.080</b>	0.001	0.000	<b>0.349</b>	0.000	0.026	0.000	0.000
2005 HA-FB RM vs. PC	0.000	0.000	<b>0.072</b>	0.004	<b>0.620</b>	0.005	<b>0.060</b>	0.005	0.001	0.000	<b>0.170</b>
2006 HA-FB RM vs. PC	<b>0.485</b>	<b>0.881</b>	<b>0.083</b>	<b>0.773</b>	<b>0.987</b>	<b>0.249</b>	0.004	<b>0.444</b>	<b>0.740</b>	0.022	<b>0.546</b>
2005 LA-FB RM vs. PC	<b>0.687</b>	<b>0.070</b>	0.005	<b>0.665</b>	<b>0.441</b>	0.017	<b>0.161</b>	0.024	0.016	0.000	0.008
2006 LA-FB RM vs. PC	0.015	0.000	0.026	0.000	0.000	0.010	<b>0.054</b>	0.000	0.003	0.030	0.001
2005 FB-RM HA vs. LA	0.000	0.000	0.010	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
2006 FB-RM HA vs. LA	0.000	0.000	0.000	0.000	0.000	0.000	<b>0.620</b>	0.000	0.000	0.001	-
2005 FB-PC HA vs. LA	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
2006 FB-PC HA vs. LA	<b>0.173</b>	0.001	0.010	0.001	0.001	0.000	0.009	0.002	0.001	0.010	0.015
2006 HA-FB RM vs. GH	0.018	<b>0.986</b>	<b>0.123</b>	<b>0.452</b>	<b>0.610</b>	<b>0.269</b>	0.025	<b>0.462</b>	<b>0.150</b>	<b>0.144</b>	0.004
2006 LA-FB RM vs. GH	0.001	<b>0.329</b>	0.033	<b>0.971</b>	<b>0.519</b>	<b>0.467</b>	<b>0.230</b>	0.002	0.002	0.002	0.028
2006 HA-FB PC vs. GH	0.010	<b>0.903</b>	<b>0.595</b>	<b>0.770</b>	<b>0.804</b>	<b>0.714</b>	<b>0.110</b>	<b>0.062</b>	0.011	0.023	<b>0.138</b>
2006 LA-FB PC vs. GH	0.006	<b>0.194</b>	0.007	<b>0.073</b>	<b>0.117</b>	<b>0.467</b>	<b>0.348</b>	0.010	0.005	<b>0.565</b>	0.001
2005 LA-FB PC vs. WR	<b>0.060</b>	0.001	<b>0.891</b>	0.000	0.000	0.030	0.016	0.001	<b>0.779</b>	0.000	0.000
2005 LA-FB RM vs. GH	0.040	<b>0.061</b>	0.016	0.023	<b>0.083</b>	0.001	<b>0.100</b>	<b>0.206</b>	<b>0.254</b>	<b>0.849</b>	0.004
TXL vs. 2005 HA-FB-RM	0.000	0.000	0.000	0.000	0.001	0.000	0.003	0.000	0.002	0.000	<b>0.137</b>
TXL vs. 2006 HA-FB-RM	0.000	0.000	0.000	0.000	0.040	0.000	0.003	0.000	<b>0.119</b>	0.000	<b>0.601</b>
TXL vs. 2005 LA-FB-RM	0.028	0.000	0.000	<b>0.116</b>	0.000	0.000	0.025	0.000	0.000	0.000	0.000
TXL vs. 2006 LA-FB-RM	0.015	0.000	0.000	0.002	0.000	0.000	0.010	0.000	0.000	0.000	0.000
PRB vs. 2005 HA-FB-RM	0.000	0.000	0.000	0.000	0.001	0.000	<b>0.530</b>	0.000	0.000	0.000	0.011
PRB vs. 2006 HA-FB-RM	0.000	0.000	0.000	0.000	<b>0.227</b>	0.000	<b>0.440</b>	0.000	<b>0.365</b>	0.000	<b>0.079</b>
PRB vs. 2005 LA-FB-RM	0.000	0.000	0.000	0.003	0.000	0.000	0.000	0.000	0.000	0.000	0.000
PRB vs. 2006 LA-FB-RM	0.000	0.000	0.000	0.050	0.000	0.000	0.044	0.000	0.000	0.000	0.000
PRB vs. TXL	0.001	0.004	0.003	0.005	0.028	0.004	0.003	0.006	0.005	0.009	0.014

Statistical analysis performed with SPSS version 13.0, equal variances assumed. **Bold** values indicate  $p > \alpha$  (0.05), means are not statistically different.

Storage effects in greenhouse (GH) or in windrow as compared with either raw or partially composed FB from 2005 or 2006 were included in the statistical comparisons. The 2005 LA-FB-PC was significantly different from LA-FB windrow-stored material for dry-basis HHV, ash, VM, FC, S, C, N, and O, but not for H or wet- or DAF-basis HHV. The 2005 LA-FB-RM differed from the 2005 LA-FB-GH material only for HHV (wet and DAF basis), ash, FC, and O.

The 2006 high-ash and low-ash FB materials were compared as a condition of storage: RM, PC, or GH. A major finding was that the GH-stored materials showed relatively few differences compared with the raw or partially composted HA or LA materials, with the marked exception of wet-basis HHV, which was significantly different for all four comparisons, along with DAF-basis HHV for LA-FB-RM or LA-FB-PC as compared with LA-FB-GH stored.

As expected, comparisons of 2005 or 2006 FB-RM (whether high- or low-ash) with Texas lignite showed significant differences ( $p \leq 0.028$ ) for HHV values (wet, dry, or DAF) and for ash, VM, FC, S, C, H, N, or O, with the following noted exceptions: 2005 HA-FB-RM (oxygen), 2006 HA-FB-RM (H and O), and 2005 LA-FB-RM (ash). Similarly, PRB coal was significantly different from HA-FB-RM and LA-FB-RM for HHV values, ash, FC, C, and N. PRB coal was not significantly different from HA-FB-RM for S (2005 and 2006), VM (2006), H (2006), or O (2006). PRB coal was significantly different from both LA-FB-RM and TXL for all the parameters compared statistically.

## SUMMARY

Research was conducted to determine the effects of feedlot surfacing materials, partial composting or storage (windrow or greenhouse), and rainfall conditions on feedlot biomass (FB) characteristics for subsequent use as biofuel. Experiments were conducted in two successive years to harvest, process, and characterize cattle feedlot biomass (FB) from open-lot cattle feeding pens (six unpaved/soil-surfaced pens and 12 paved/fly-ash surfaced pens) for bio-fuel properties related to combustion (reburn or co-firing) or gasification, including mixtures with coal or lignite. Standard analyses included proximate, ultimate, higher heating value, elemental analysis of ash, and selected minerals (e.g., phosphorus, sulfur, chlorine, and mercury), with bulk density determined on uncomposted FB materials in the first year.

FB was harvested following a relatively high rainfall winter-spring period and was windrow-composted for a limited period. The experiment was repeated the next year following a relatively dry winter-spring season. Harvested FB was partially composted in windrows, and bulk samples were stored for 9 to 12 months in windrows or a greenhouse to determine storage effects.

Bulk density was determined for uncomposted 2005 harvested FB only. Using a modified version of ASABE Standard S269.4 (*ASABE Standards*, 2011), bulk density of 2005 LA-FB averaged  $512 \pm 5 \text{ kg m}^{-3}$  ( $32.0 \pm 0.3 \text{ lbs ft}^{-3}$ ), as compared to  $747 \pm 14 \text{ kg m}^{-3}$  ( $46.7 \pm 0.9 \text{ lbs ft}^{-3}$ ) for HA-FB. By comparison, ASTM Standard D1895 (ASTM, 2003) yielded bulk density values of  $430 \pm 0.5 \text{ kg m}^{-3}$  ( $26.8 \pm 0.03 \text{ lbs ft}^{-3}$ ) and  $651 \pm 11 \text{ kg m}^{-3}$  ( $40.6 \pm 0.7 \text{ lbs ft}^{-3}$ ) for the 2005 LA-FB and HA-FB, respectively.

The HA-FB harvested from traditional unpaved soil-surfaced feedlot pens was contaminated with high ash content (58.7%  $\pm$  1.7% d.b. wet-year harvested or 45.2%  $\pm$  3.1% d.b. dry-year harvested) due to hoof-entrainment of underlying soil. By contrast, the LA-FB from fly-ash paved pens, which prevented soil entrainment, had about one-third the ash content (20.2%  $\pm$  1.1% and 13.6%  $\pm$  0.9% d.b. in wet and dry years, respectively).

The effect of antecedent seasonal rainfall on HHV was significant. Dry year (2006) harvested HA-FB and HA-FB-PC had higher HHV values compared with wet year (2005) harvested HA-FB and HA-FB-PC. The HHV values for LA-FB were significantly affected by harvest year for raw FB (dry and DAF basis) and for LA-FB-PC (wet and DAF basis). As-harvested uncomposted mean HHV values for HA-FB were 6,304  $\pm$  78 kJ kg<sup>-1</sup> and 8,190  $\pm$  488 kJ kg<sup>-1</sup> (2,710  $\pm$  34 BTU lb<sup>-1</sup> and 3,521  $\pm$  210 BTU lb<sup>-1</sup>) w.b. for the wet year and dry year, respectively. For LA-FB, the as-harvested, uncomposted HHV was 13,407  $\pm$  342 kJ kg<sup>-1</sup> and 13,224  $\pm$  338 kJ kg<sup>-1</sup> (5,764  $\pm$  147 BTU lb<sup>-1</sup> and 5,685  $\pm$  145 BTU lb<sup>-1</sup>) w.b. for the wet year and dry year, respectively. On a DAF basis, the HHV for HA-FB averaged 19,072  $\pm$  762 kJ kg<sup>-1</sup> and 20,566  $\pm$  115 kJ kg<sup>-1</sup> (8,200  $\pm$  327 BTU lb<sup>-1</sup> and 8,842  $\pm$  50 BTU lb<sup>-1</sup>) for 2005 and 2006, respectively. After partial composting (HA-FB-PC), the HHV averaged 17,868  $\pm$  394 kJ kg<sup>-1</sup> and 20,014  $\pm$  398 kJ kg<sup>-1</sup> (7,682  $\pm$  169 BTU lb<sup>-1</sup> and 8,604  $\pm$  171 BTU lb<sup>-1</sup>) for 2005 and 2006, respectively. By comparison, the HHV for LA-FB from paved feed pens averaged 21,072  $\pm$  30 kJ kg<sup>-1</sup> and 21,627  $\pm$  112 kJ kg<sup>-1</sup> (9,059  $\pm$  13 BTU lb<sup>-1</sup> and 9,298  $\pm$  48 BTU lb<sup>-1</sup>) on a DAF basis for 2005 and 2006, respectively, before composting. After partial composting (LA-FB-PC), the HHV averaged 20,773  $\pm$  88 kJ kg<sup>-1</sup> and 21,195  $\pm$  184 kJ kg<sup>-1</sup> (8,931  $\pm$  38 BTU lb<sup>-1</sup> and 9,112  $\pm$  79 BTU lb<sup>-1</sup>) DAF for 2005 and 2006, respectively.

Partial composting in windrows slightly increased the ash content and reduced the HHV compared with uncomposted FB. For 2005 harvested FB, partial composting for 51 to 55 days reduced wet basis (as received) HHV by 20% to 5,207  $\pm$  114 kJ kg<sup>-1</sup> (2,239  $\pm$  49 BTU lb<sup>-1</sup>) at 17% moisture for HA-FB and by 2% to 13,267  $\pm$  446 kJ kg<sup>-1</sup> (5,704  $\pm$  192 BTU lb<sup>-1</sup>) at 19.6% moisture for LA-FB. For 2006 harvested manure, final moisture contents were elevated to 30% to 43% (above precomposted conditions). At the end of the 91-day composting period, the higher moisture may have contributed to the large reduction in HHV (w.b.) to 7,755  $\pm$  850 kJ kg<sup>-1</sup> (3,334  $\pm$  365 BTU lb<sup>-1</sup>) for HA-FB-PC and to 9,454  $\pm$  1560 kJ kg<sup>-1</sup> (4,064  $\pm$  671 BTU lb<sup>-1</sup>) for LA-FB-PC.

Long-term (9 to 12 months) storage reduced the moisture to 17.6% in a windrow or 10.3% to 12.7% w.b. in a greenhouse, maintained or increased the ash content, and may have contributed to ongoing reduction of volatile solids and HHV by up to 20%. The effect of greenhouse storage/drying to 7.7% to 13.4% w.b. for both FB harvest years appeared to have preserved HHV values. Partially composted FB stored in a greenhouse was superior in HHV to

windrow-stored manure for both 2005 LA-FB-PC and 2005 LA-FB-RM; similar windrow vs. greenhouse data were not available for 2006 FB.

A frequency distribution was developed for DAF-basis HHV, which ranged from 17,000 to 22,000 kJ kg<sup>-1</sup> (7,296 to 9,442 BTU lb<sup>-1</sup>) with an average of 20,500 kJ kg<sup>-1</sup> (8,798 BTU lb<sup>-1</sup>). The mode of the high-ash FB was 20,500 kJ kg<sup>-1</sup> (8,798 BTU lb<sup>-1</sup>), while the mode of the low-ash FB was 21,500 kJ kg<sup>-1</sup> (9,227 BTU lb<sup>-1</sup>). All of the low-ash FB values were between 20,000 to 22,000 kJ kg<sup>-1</sup> (8,584 to 9,442 BTU lb<sup>-1</sup>) DAF, while 72% of the high-ash FB values were between 19,000 to 21,000 kJ kg<sup>-1</sup> (8,155 to 9,013 BTU lb<sup>-1</sup>) DAF.

Direct comparisons were made between FB, TXL, and PRB coal as to HHV and chemical properties. Moisture contents for TXL and PRB coal (38.34%  $\pm$  0.34% w.b. and 32.88%  $\pm$  0.36% w.b., respectively) were higher than for the FB materials. Ash contents were much lower for the PRB coal (8.40%  $\pm$  3.11% d.b.) compared with 18.59%  $\pm$  0.85% d.b. for TXL. Ash contents for TXL were within the range of ash contents for LA-FB from fly-ash paved pens and much lower than for HA-FB. HHV was higher for both TXL and PRB coal compared with feedlot biomass, on a wet basis (as received), dry basis, and DAF basis. Comparing mean HHV values for FB (both project years) with TXL and PRB coal produced the following rankings:

- Wet basis (kJ kg<sup>-1</sup>): HA-FB (7,247) < LA-FB (13,316) < TXL (14,289) < PRB coal (18,196).
- Dry basis (kJ kg<sup>-1</sup>): HA-FB (9,564) < LA-FB (17,753) < TXL (23,172) < PRB coal (27,114).
- DAF basis (kJ kg<sup>-1</sup>): HA-FB (19,819) < LA-FB (21,350) < TXL (28,462) < PRB coal (29,596).

Heating value on a dry ash-free (DAF) basis averaged 20,984 kJ kg<sup>-1</sup> (9,022 BTU lb<sup>-1</sup>) for LA-FB-PC and 18,941 kJ kg<sup>-1</sup> (8,143 BTU lb<sup>-1</sup>) for HA-FB-PC for both harvest years.

## CONCLUSIONS

From the data presented, the following conclusions are warranted:

- Bulk density of 2005 LA-FB was approximately two-thirds that of HA-FB. Bulk densities obtained using a modified version of ASABE Standard S269.4 (*ASABE Standards*, 2011) were 15% to 19% higher compared with those obtained using ASTM Standard D1895 (ASTM, 2003).
- Statistically significant differences were determined between HA-FB and LA-FB for both years for the following parameters (dry matter basis): ash, volatile matter, fixed carbon, total carbon, heating value (HHV), and nitrogen.
- Ash content of LA-FB averaged approximately one-third that of HA-FB for both years and was similar to that of Texas lignite.
- As-harvested 2006 HA-FB and LA-FB showed statistically improved differences in biofuel characteristics in terms of lower ash contents and higher values of HHV,

volatile matter, fixed carbon, and total carbon than for the counterpart 2005 manure. These differences may be attributed to drier winter-spring conditions that preceded the May-June 2006 manure harvest, which likely reduced hoof-entrainment of soil from below the manure pack in the HA-FB and may have reduced rates of *in situ* decomposition.

- As-harvested LA-FB had significantly higher HHV than HA-FB on a dry basis and DAF basis for both harvest years.
- HHV before composting on wet (as received) and dry basis averaged 85% higher for manure from paved (LA-FB) pens compared with soil-surfaced (HA-FB) pens. Differences were more pronounced following the wet year (2005) compared with the dry year (2006) FB harvest.
- Elemental analysis showed that ash from LA-FB was higher than ash from HA-FB in Ca, P, Cl, K, Mg, Na, and S, but it was lower in Si, Al, Ti, and Fe before composting and after partial composting. Metal contents appeared to be similar for both high-ash and low-ash FB. Sufficient data were not available to determine statistical differences.
- Greenhouse storage of the 2005 harvested LA-FB for 9 to 12 months preserved fuel quality of both raw manure and partially composted manure better than continuous storage of LA-FB-PC in a static windrow. Greenhouse storage of 2005 and 2006 HA-FB, HA-FB-PC, LA-FB, and LA-FB-PC reduced moisture content to near 10% w.b. (7.7% to 13.4% range) with relatively small changes in dry basis values of ash, HHV, FC, VM, total C, H, O, and S, as corroborated by statistical analysis.
- The HHV of feedlot biomass on a dry ash-free (DAF) basis varied depending on ash content and perhaps other variables that were beyond the scope of this study. For high-ash feedlot biomass, in this study, the HHV-DAF values had a mode of 20,500 kJ kg<sup>-1</sup> (8,798 BTU lb<sup>-1</sup>), as compared to a mode of 21,500 kJ kg<sup>-1</sup> (9,227 BTU lb<sup>-1</sup>) for the low-ash FB harvested from fly-ash surfaced feedpens.
- PRB coal was significantly different from both LA-FB-RM and Texas lignite for all the parameters compared statistically. Texas lignite and PRB coal were significantly different from HA-FB-RM and LA-FB-RM for HHV values, ash, FC, C, and N, with the exception that Texas lignite was not significantly different from 2005 LA-FB-RM for ash content.

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