Poultry Litter Physiochemical Characterization Based on Production Conditions for Circular Systems

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Poultry litter is a useful product as a fertilizer, energy feedstock for thermochemical conversion, and a precursor for synthesis of adsorbents and catalysts. Detailed characterization of baseline properties is necessary for enhanced environmental and economic utilization of this valuable resource. Baseline physicochemical characterization was carried out at two broiler production facilities (Arkansas, PL1, and North Carolina, PL2). Greater concentrations of inorganic nitrogen, phosphorus, and potassium were obtained for PL1, suggesting greater nutrient value compared to PL2. PL2 had greater carbon content and water-holding capacity than PL1. X-ray photoelectron spectroscopy (XPS) of PL1 and PL2 indicated a similarity between litters in terms of the presence of carbon, nitrogen, and oxygen bonds. Both poultry litters had oxygen, nitrogen, sulfur, and phosphorous functional groups, as confirmed by infrared spectroscopy. Time of flight - secondary ion mass spectroscopy of negative ions also indicated similarity of the surface charge distribution between PL1 and PL2. Overall, poultry litters evaluated had similar surface chemistries, with nutrient composition varying based on rearing conditions, which has implications for downstream use in thermochemical conversion and other value-added products.

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INTRODUCTION

Over 9 billion broilers (meat chicken) are produced in the U.S. annually (USDA-NASS 2021). Production of broilers in confined animal production facilities results in a large quantity of litter, which is comprised of manure, bedding material, and spilled feed. Variable composition of poultry litter occurs because of the type of bird reared (broilers, layers, or turkeys), source and thickness of bedding material used (*e.g.*, rice husk, wood shavings, straw, sawdust, peanut hulls, and other agricultural residues), frequency of cleanout, and handling and storage of the produced litter (Bolan *et al.* 2010; Ashworth *et al.* 2020). It is estimated that nearly 14 million tons of litter is produced annually just from broiler production units in the U.S. (Ashworth *et al.* 2020). Because poultry litter is loaded with high amounts of nutrients essential for plant growth, such as nitrogen (N), phosphorus (P), and potassium (K) (Ashworth *et al.* 2020), it has traditionally been applied to agricultural land as an organic fertilizer (Sharpley *et al.* 2009). Application of poultry litter to croplands is shown to improve soil fertility, earthworm and microbial communities, as well as soil physical and hydrological properties such as aggregate stability, infiltration rate, and hydraulic conductivity (Adeli *et al.* 2009; Ashworth *et al.* 2018; He *et al.* 2019; Feng *et al.* 2021). However, long-term land application of poultry litter has been shown to accumulate nutrients and trace metals in soil (Daigh *et al.* 2009), and through runoff and leaching it has the potential to impair surface and groundwater quality (Sharpe *et al.* 2004; Bolan *et al.* 2010; McMullen *et al.* 2014). Additionally, land application of poultry litter is also associated with other environmental concerns such as spread of pathogens, air pollution, greenhouse gas emissions, and production of phytotoxic substances (Kelleher *et al.* 2002). This has resulted in the implementation of stringent regulatory measures for nutrient management and land application of manure (Sharpley *et al.* 2009).

Because of excessive generation of poultry litter, more is often produced than can be sustainably applied to croplands; therefore, alternative uses are vital for the sustainability of the poultry industry. Several studies have shown that agricultural wastes such as poultry litter can be converted into value-added products, the most common being conversion to biochar for use as a pathogen-free soil amendment (Cantrell *et al.* 2012; Song and Guo 2012; Novak *et al.* 2012; Katuwal *et al.* 2022) to improve and maintain soil fertility, soil quality, water-holding capacity, and soil carbon sequestration (Chan *et al.* 2008; Novak *et al.* 2009), or remediation of contaminants from soil and water (Lima and Marshall 2005; Guo *et al.* 2010; Lima *et al.* 2015). Alternative utilization methods of this valuable resource include its use as a biomass energy source via combustion for heat and energy (Lynch *et al.* 2013), biogas production by anaerobic digestion (Beausang *et al.* 2020; USEPA 2022), and generation of electricity (Dagnall *et al*. 2000) with lower environmental impact compared to land application of raw poultry litter with respect to pathogens, pollutants, and emissions of volatile gases (NH_3, N_2O, and NO_x gases) (Billen *et al.* 2015).

Mostly, poultry litter and its derived products are characterized and discussed for their potential use for a specific purpose. Knowledge of litter properties, such as moisture content, ash content, amount of volatile matter, energy density, calorific value, fixed carbon, and elemental contents are important for their effective and economic utilization (Khan *et al.* 2009). One of the challenges related to determining utilization potential of poultry litter relates to the variation of its physiochemical composition, because of the use of different biomass as bedding materials, the variation in management, storage, and handling of poultry litter (Bolan *et al.* 2010; Crippen *et al.* 2016). Detailed characterization of properties of poultry litter can aid in selection and/or development of technology or processes to achieve higher yield from the resources at hand. For instance, physical properties including bulk density, particle density, porosity, compressibility, particle size, and particle size distribution are relevant to the designing of equipment for handling, storage, and processing of poultry litter (Bernhart *et al.* 2007; Bernhart and Fasina 2009). Properties, such as moisture content, ash content, volatile matter, and energy density, are reported to affect the calorific value and corrosive properties of biomass resources intended for use as energy sources (Bolan *et al.* 2010; Billen *et al.* 2015; Santos Dalólio *et al.* 2017).

Therefore, the overall objective of this study was to provide a detailed characterization of physical and chemical properties of poultry litter obtained from two commercial facilities representative of two major poultry producing states (Arkansas and North Carolina) in U.S. The properties of poultry litter from the two commercial facilities are discussed and compared in relation to their value as a soil amendment and biofeedstock source. The authors surmise that such a detailed characterization of poultry litter will aid in the comparison and selection of application of this important feedstock.

EXPERIMENTAL

Collection of Poultry Litter

Poultry litter obtained from a production facility in Arkansas (PL1) and North Carolina (PL2) were evaluated. Production conditions in Arkansas consisted of pens (2.1 × 1.8 m^2, 50 broiler chicks per pen, and reared for 42 d) in Fayetteville, Arkansas. Pine wood shaving was used as the bedding material (17.5 kg per pen, depth of 5 cm) over concrete floors. The litter was obtained after 3 flocks of birds were reared, during which the same bedding material was reused without addition of new material. The broiler's feed during the rearing period of 42 days contained corn (64.2%), soybean meal (27.7%), 50% meat, and bone meal (2.5%), poultry oil (2.65%), sodium chloride (0.31%), sodium bicarbonate (0.05%), limestone (0.74%), dicalcium phosphate (1%), vitamins, amino acids, trace metals, xylanase, and phytase (Anderson *et al.* 2021). Thus, the litter was a mixture of manure, bedding material, spilled feed, and other wastes from the birds. Litter obtained from different areas within a pen and replicate pens (4 pens) were homogenized, and subsamples were refrigerated until analyzed. Similarly, the litter samples from North Carolina were collected from several locations in a commercial farm stocked with birds approximately 1 bird per 0.30 m and fed with commercial feed (about 65 to 70% ground corn and 20 to 25% soybean meal, with the remaining balance composed of fat, salt, vitamins, minerals, dicalcium phosphate, and amino acids) with pine shavings as the bedding material that was less than two years old while the caked litter was cleaned out after every flock (about 9 weeks). All samples were refrigerated until they were analyzed.

Physical Properties

Moisture content was determined gravimetrically after drying litter samples at 105 °C for 24 h. The pH and electrical conductivity (EC) were determined in a 1:10 litter and water mixture with a pH electrode and a conductivity meter, respectively. Bulk density was determined by filling a 10-mL tube successively with about 1 mL of oven-dried sample and tapping after each addition to a constant minimum volume as described by Lima and Marshall (2005). The ratio of oven-dry litter weight to the volume of packed litter provided the bulk density. Water-holding capacity (WHC) of litter was determined on ground and sieved (< 2-mm) litter samples that were uniformly mixed and packed in PVC collars (about 100 cm^3). The samples were irrigated until saturated flow occurred, following which they were placed in a pressure plate extractor (Soilmoisture Equip. Corp., Santa Barbara, CA, USA) under a pressure of 33 kPa for 2 to 3 days until a constant moisture content was obtained as described by Ashworth *et al.* (2014). The WHC was calculated as amount of moisture retained at 33 kPa per unit dry mass of litter.

Nutrient and Elemental Composition

Total carbon (TC) and total nitrogen (TN) were determined by combustion of litter using a Vario Max CN analyzer (Elementar Americas Inc., Ronkonkoma, NY, USA). Nitrate-nitrogen (NO_3-N), ammonium-nitrogen (NH_4-N), and soluble reactive phosphorus (SRP) were determined on 1:10 litter/water extraction following filtration through a 0.45-µm filter paper (Self-Davis and Moore 2000) by colorimetric analysis on a Skalar auto-analyzer (Skalar, Analytical B.V., AA Breda, Netherlands). Nitrate-nitrogen was analyzed by the Cd-reduction method according to American Public Health Association Method 418-F (APHA 1992), NH_4-N was analyzed by the salicylate-nitroprusside USEPA Method 351.2 (USEPA 1979), and SRP by the Murphy and Riley (1962) method. Organic N was calculated as the difference between the total N and sum of the inorganic N forms, NH_4-N, and NO_3-N. Total metals (Al, As, Ca, Cd, Co, Cr, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, Se, Ti, and Zn) were determined on oven dried-litter samples by inductively coupled optical emission spectroscopy (ICP-OES) on an Agilent 5110 ICP-OES (Agilent Technologies, Santa Clara, CA, USA) after digesting litter samples with HNO_3 and H_2O_2 (Zarcinas *et al.* 1987). Soluble metals (Al, As, Ca, Cd, Co, Cr, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, Se, Ti, and Zn) were extracted using fresh litter and a 1:10 (litter:water) extraction ratio according to Self-Davis and Moore (2000) and analyzed by ICP-OES.

Surface Chemical Characterization of Litter

Acid values of poultry litters were determined using the procedure described in Kolar and Jin (2019). Briefly, 0.4 g of litter samples were added to 20 mL of deionized water and allowed to equilibrate for 24 h. Subsequently, the litter was separated from the solution *via* filtration and the pH of the solution was determined as its acid value. Poultry litter surfaces were analyzed by X-ray photoelectron spectroscopy (XPS) in a SPECS XPS system with a PHOIBOS 150 analyzer using Mg Kα radiation under a pressure of about 3 x 10^{-10} mbar. To identify and quantify chemical functional groups present on surfaces, time-of-flight-secondary ion mass spectroscopy (ToF-SIMS) was used. The ToF-SIMS analysis was conducted by ION ToF – SIMS^5 using Bi^+ ion gun and ToF mass analyzer. Poultry litter samples were subjected to 1.2 x 10^{-8} mbar pressure. The data were collected in negative ion modes for complete characterization. Data were also corroborated with additional infrared (IR) spectra analysis using a Bruker Platinum ATR spectrometer (Bruker, Billerica, MA, USA). The IR spectral data were recorded within a range of 400 to 4500 cm^{-1}.

Data Analysis

Physiochemical composition of litter obtained from the two production facilities were compared using Welch's t-test in SAS v9.4 (SAS Institute, Cary, NC, USA) at a probability level of 0.05. Data collected from XPS analysis were deconvoluted *via* XPSPeak41 software for the peaks associated with carbon, oxygen, and nitrogen. The spectra were fitted with Shirley background. The deconvoluted data were plotted using Origin 2021b software. The IR raw data were processed by plotting them using Origin 2021b software (OriginLab Corp., Northampton, MA, USA).

RESULTS AND DISCUSSION

Composition of Poultry Litter

As a soil amendment

The composition of poultry litter obtained from the two facilities is presented in Table 1. The two litter sources varied considerably in their composition and nutrients. Given the wide variations in poultry production parameters, litter management, handling, and storage conditions, variation in the composition and content of nutrients in poultry litter among poultry production facilities is common (Ashworth *et al.* 2020). Litter from both facilities had a moisture content > 30%, with PL1 having greater moisture (about 39.1%) than PL2 (30.2%). Moisture content of litter can affect the handling of litter during storage and transporting (Bernhart *et al.* 2007; Bernhart and Fasina 2009), as well as ammonia losses from litter (Miles *et al.* 2011) and is an important parameter when calculating nutrient content and rate of nutrients applied.

Litter pH is an important factor driving ammonia volatilization. At pH values greater than 8, as observed for both litters in this study, volatilization of NH_4-N to ammonia gas occurs, thus decreasing N value of litter during or after land surface applications (Sharpe *et al.* 2004; Ashworth *et al.* 2020). However, high pH can also be beneficial in terms of increasing soil pH, thus acting as a liming agent in acidic soils of the southeastern U.S. (He *et al.* 2019). Poultry litter EC is representative of the concentration of soluble salts in litter and was higher for PL1 than PL2 (Table 1).

Table 1. Poultry Litter Properties (Mean ± Standard Deviation) Obtained from Facilities in Arkansas (PL1) and North Carolina (PL2)

Litter Properties [a]	PL1 [b]	PL2 [b]
Moisture content (%)	39.14 ± 0.44 a [c]	30.2 ± 0.67 b
pH (H_2O)	8.86 ± 0.02 a	8.49 ± 0.02 b
EC (mS cm^{-1})	9.59 ± 0.06 a	6.74 ± 0.11 b
Bulk density (g cm^{-3})	0.39 ± 0.01 b	0.42 ± 0.01 a
WHC (g g^{-1})	1.24 ± 0.07 b	1.95 ± 0.02 a
Organic-N (%, w.b.)	1.61 ± 0.17 b	2.29 ± 0.04 a
NH_4-N (g kg^{-1}, w.b.)	6.12 ± 0.14 a	1.84 ± 0.02 b
NO_3-N (mg kg^{-1}, w.b.)	27.23 ± 11.6 a	3.8 ± 0.05 b
TN (%, w.b.)	2.22 ± 0.19 a	2.47 ± 0.04 a
TC (%, w.b.)	20.56 ± 2.34 b	32.32 ± 0.49 a
C/N	9.35 ± 1.83 b	13.1 ± 0.28 a
P (%, d.b.)	2.16 ± 0.07 a	1.63 ± 0.07 b
K (%, d.b.)	3.67 ± 0.06 a	3.30 ± 0.15 b
Ca (%, d.b.)	3.25 ± 0.17 a	2.15 ± 0.04 b
Mg (%, d.b.)	0.61 ± 0.05 a	0.66 ± 0.03 a
S (%, d.b.)	0.94 ± 0.02 a	0.55 ± 0.02 b
Al (mg kg^{-1}, d.b.)	309.17 ± 14.15 b	586.67 ± 67.14 a
As (mg kg^{-1}, d.b.)	0.68 ± 0.13 b	1.3 ± 0.28 a
Cd (mg kg^{-1}, d.b.)	0.65 ± 0.22 a	0.30 ± 0.0 b
Co (mg kg^{-1}, d.b.)	0.77 ± 0.03 b	0.93 ± 0.06 a
Cr (mg kg^{-1}, d.b.)	6.27 ± 0.43 a	5.42 ± 0.40 a
Cu (mg kg^{-1}, d.b.)	530 ± 21.79 b	636.67 ± 49.33 a
Fe (mg kg^{-1}, d.b.)	274 ± 56.31 a	163.17 ± 62.54 b
Mn (mg kg^{-1}, d.b.)	608.33 ± 17.56 a	441.5 ± 22.11 b
Mo (mg kg^{-1}, d.b.)	5.82 ± 0.06 a	3.65 ± 0.48 b
Na (%, d.b.)	0.63 ± 0.01 a	0.69 ± 0.05 a
Ni (mg kg^{-1}, d.b.)	17.43 ± 0.33 a	8.83 ± 0.35 b
Pb (mg kg^{-1}, d.b.)	0.72 ± 0.1 a	0.73 ± 0.19 a
Se (mg kg^{-1}, d.b.)	4.03 ± 0.88 a	1.75 ± 0.23 b
Ti (mg kg^{-1}, d.b.)	0.18 ± 0.16 a	0.38 ± 0.19 a
Zn (mg kg^{-1}, d.b.)	606.67 ± 17.56 b	680 ± 40 a

[a] Organic-N = Total N – (NH_4-N + NO_3-N); EC, electrical conductivity; d.b., dry weight basis; w.b., wet weight basis or ‘as-is’ basis. [b] PL1 and PL2 refer to poultry litter obtained from production facilities in Arkansas and North Carolina, respectively. Different letter for a variable along a row represents significant difference between PL1 and PL2 at $p \leq 0.05$.

Total C constituted the greatest fraction of both litter sources, with about 34% and 46% of the total dry matter in PL1 and PL2, respectively. Because of the high content of total C in litter, its land application may improve soil quality and crop production by increasing soil organic carbon and consequently enhancing soil aggregation and aggregate stability, increasing soil porosity and WHC, improving hydraulic properties of soil, and increasing soil microbial community (Adeli *et al.* 2007; Yang *et al.* 2019; Feng *et al.* 2021).

Bulk density of each litter was lower than 0.50 g cm^{-3}, with greater density for PL2 than PL1. While lower bulk density is a favorable characteristic when used as a soil amendment, as it can improve hydraulic properties; however, this would impact transportation costs because of the large volume associated with loose material. Litter collected from both the facilities had high WHC. The average values were 1.24 and 1.95 g g^{-1} in PL1 and PL2, respectively.

The amount of total N in the two litters were similar for PL1 and PL2 (2.22% and 2.47%, respectively). Organic-N constituted more than 70% and 90% of the total N present in PL1 and PL2, respectively, and the inorganic-N (NH_4-N + NO_3-N) constituted approximately 28% and 7% of the total N in PL1 and PL2, respectively. While the inorganic N forms are readily available for plant use, the organic fraction has to be mineralized before being plant available, suggesting when land applied, PL1 would provide more N in readily available forms than PL2.

The amount of P averaged 2.16% and 1.63% for PL1 and PL2, of which, approximately 14% and 8% constituted water extractable P for the two litters, respectively. Potassium content was 3.7% and 3.3% for PL1 and PL2, respectively. Table 2 shows major nutrient poultry litter fertilizer values. While N and K_2O values were similar for the litter from the two production facilities, PL1 had greater P_2O_5 than PL2. In addition to N, P, and K, both litter sources contained substantial quantities of Ca, Mg, and S, and other micronutrients such as Cu, Fe, Mo, Mn, Ni, and Zn, which are essential for plant growth. While supplying essential plant nutrients and improving soil quality, over-applications of poultry litter long-term may be deleterious to water quality because of P and N losses *via* leaching and runoff (Kingery *et al.* 1994; Daigh *et al.* 2009; He *et al.* 2019). Land application following best nutrient management practices can support its use as an excellent fertilizer for forages and row crops, while minimizing environmental concerns related with land application of poultry litter and improving soil health and quality (Amorim *et al.* 2020; Ylagan *et al.* 2021).

Table 2. Total Nitrogen (N), Phosphorus (P) and Potassium (K) in Poultry Litter (Mean ± Standard Deviation)

Poultry Litter	N (kg Mg^{-1}) [a]	P_2O_5 (kg Mg^{-1})	K_2O (kg Mg^{-1})
PL1 [b]	22.2 ± 1.9 a [c]	30.03 ± 1.10 a	26.79 ± 0.65 a
PL2 [b]	24.7 ± 0.4 a	26.07 ± 0.86 b	27.62 ± 1.06 a

[a] N, P_2O_5, and K_2O are presented in "as-is" basis. [b] PL1 and PL2 refer to poultry litter obtained from production facilities in Arkansas and North Carolina, respectively. [c] Different letter for a variable along a row represents significant difference between PL1 and PL2 at $p \leq 0.05$.

As a biomass energy source

Poultry litter could be used as a source of biomass energy through thermochemical conversion processes, such as direct combustion, gasification, and pyrolysis. The main characteristics for determining the utilization of poultry litter as a source of biomass energy include moisture content, energy density, calorific value, volatiles content, ash content, the fixed carbon content, chemical analyses, and elemental contents (Khan *et al.* 2009; Dalólio *et al.* 2017). The concentrations of volatiles, carbon, and hydrogen contents, sulfur concentration, and moisture content determine the energy requirement during the thermochemical processes such as gasification and pyrolysis. Biomass with low moisture, high volatiles, carbon, and hydrogen content, and low sulfur concentration requires lower temperatures and thus energy during the thermochemical processes and are therefore favorable (Abelha *et al.* 2003; Dalólio *et al.* 2017).

When intended for use as a feedstock or as a precursor for biochar or energy production, PL2 with lower moisture content (30.2%) was better suited than PL1 (39.1%), as lower moisture content of litter negatively affects the energy requirement of the system for combustion, influences the initial ignition capability, the degree of combustion, and consequent release of carbon monoxide to the environment (Dávalos *et al.* 2002). Greater moisture content in PL1 than PL2 also suggests that PL1 would require additional energy and resources for drying before transport, use as an energy feedstock or during the thermochemical conversion process. Moisture content is a major factor determining the price paid to poultry producers for poultry litter utilization in power plants (Dagnall *et al*. 2000). Greater moisture in PL1 compared to PL2 could result from differences in poultry litter management at the two production facilities. In Arkansas, caked litter was not removed but the litter was tilled between each flock (Anderson *et al.* 2021), whereas in North Carolina caked litter was removed after each flock.

One of the environmental impacts associated with the utilization of poultry litter as a source of biomass energy is emission of nitric oxides (NO, NO_2), with N concentration (wt%, d.b.) > 0.6 and sulfur oxides (SO_2, SO_3) emissions (wt%, d.b.) > 0.2 (Obernberger *et al*. 2006). Greater concentrations of N and S in both PL1 (N: 3.65 % d.b. and S: 0.94 % d.b.) and PL2 (N: 3.54 % d.b. and S: 0.55 % d.b.) suggests appropriate measures and possible technologies [as outlined in Obernberger *et al*. (2006)] should be adopted for reducing the emissions associated with the use of these resources for energy generation.

Lync *et al.* (2003) reported the energy content of fuel, expressed as higher heating value (HHV), between 16.49 and 20.4 GJ Mg^{-1} (d.b.) of poultry litter with moisture content between 18.7 and 51.8%. Lower bulk density of PL1 (Table 1) is associated with lower energy density of PL1 compared to PL2 which would impact the cost associated with transportation of PL1 for off-farm or centralized systems. Lower moisture content, greater C content, and bulk density, lower sulfur content suggests as PL2 being a better option as a source of biomass energy in terms of energy content, economy, and environmental concerns than PL1.

Surface Chemical Characterization of Litter

Surface chemical analyses of PL1 and PL2 provide valuable information of surface chemical compositions and bonds across sources of poultry litter. The acid value determination helps to understand the surface pH. The XPS, ToF-SIMS, and IR analyses generate a comprehensive and detailed view of the surface bonds and chemical compositions. Such analyses may help drive end uses of poultry litter.

Acid value

Tiquia and Tam (2002) reported pH of poultry litter collected from different locations of the pile to be between 8.18 and 8.33. Similar observations were also made by de Souza *et al.* (2019), where the pH of uncomposted poultry litter was 8.35. The slightly basic nature of litter suggests that litter-derived products, when applied as soil amendments, could serve as liming agents.

Table 3. Acid Values of Poultry Litter Collected from Arkansas (PL1) and North Carolina (PL2)

Type of Poultry Litter	Mean Acid Value	Standard Deviation	Standard Error
PL1 [a]	9.00	0.04	0.023
PL2 [a]	8.92	0.23	0.13

[a] PL1 and PL2 refer to poultry litter collected from Arkansas and North Carolina production facilities, respectively.

Infrared Analysis

Figure 1 represents the infrared spectra of PL1 and PL2. The spectra indicate the presence of sulfur, nitrogen, phosphorous, and oxygen functional groups in both poultry litters. Table 4 presents the functional groups associated with the different peaks detected. The absorption band at 3274 cm^{-1} corresponds to hydroxyl stretching from alcohol, amide, and carboxyl groups. Bands at 1633 cm^{-1} and 1547 cm^{-1} were likely because of cyclic amide and N = N stretching, respectively. Bands around 1030 cm^{-1} and 560 cm^{-1} were attributed to SO_3H and P-O groups, respectively, which confirm the presence of sulfur and phosphorous in poultry litter. The existence of the same functional groups in PL1 and PL2 indicates the similarity of poultry litter surfaces in terms of chemical nature irrespective of their sources. de Souza *et al.* (2019) recorded the infrared spectra of raw poultry litter and reported the existence of hydroxyl, amide, and aromatic groups.

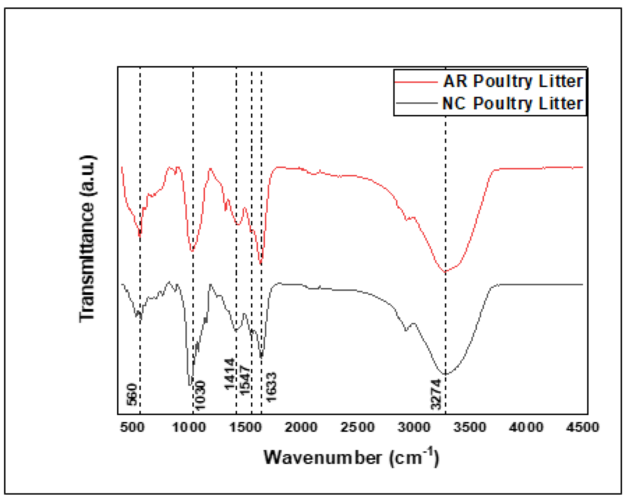

Fig. 1. Infrared spectra of poultry litter collected from North Carolina (NC) and Arkansas (AR)

Table 4. Surface Functional Groups of the Litters Collected from North Carolina and Arkansas

Peaks (cm^{-1})	Functional Groups	Reference
3274	O – H Stretching	Biniak *et al.* (1997)
1633	Cyclic amide	Shafeeyan *et al.* (2010)
1547	N = N Stretching	Kim *et al.* (2007)
1414	O – H bending	Zhang *et al.* (2019)
1030	SO_3H group	González *et al.* (2017)
560	P-O asymmetric stretching Sulfate	Sáez Del Bosque *et al.* (2014); Wang *et al.* (2017); Zhang *et al.* (2019)

X-ray photoelectron spectroscopy analysis

High resolution X-ray photoelectron spectroscopy (XPS) spectra of PL1 and PL2 gave additional information of surface functionalities. The surface elements analyzed were carbon (C1s), nitrogen (N1s), and oxygen (O1s). Table 5 illustrates the elemental compositions of C1s, N1s, and O1s on poultry litter surfaces. Each elemental composition was deconvoluted into components' peaks and expressed as their relative contents, as shown in Table 6 and Figs. 2 and 3. The analysis provides chemical states and identifies which individual element was present. C1s spectra were deconvoluted into three component peaks (C1, C2, and C3): C1 (284.8 to 285 eV), which indicates the presence of C-C; C2 (286.2 to 286.7 eV) indicates the presence of C-O; C3 (287.9 to 288.2), thereby indicating the presence of C=O (Chen *et al.* 2020). Therefore, carbon was present in PL1 and PL2 as C-O, C=O, and C-C. Cao *et al.* (2020) observed similar results where the presence of C-O, C=O, and C-C was identified in poultry litter.

Table 5. Elemental Composition of Arkansas (PL1) and North Carolina (PL2) Poultry Litters Obtained by XPS

Sample	C1s (%)	N1s (%)	O1s (%)
PL1 [a]	70.5	6.4	22.0
PL2 [a]	72.2	5.1	22.7

[a] PL1 and PL2 refer to poultry litter collected from Arkansas and North Carolina production facilities, respectively.

Table 6. Relative Speciation of the Elemental Composition of Arkansas (PL1) and North Carolina (PL2) Poultry Litters Obtained by XPS

Peaks	Components	PL1 [d] (%)	PL2 [d] (%)
C1s	C1 [a]	67.99	77.06
	C2 [a]	27.69	8.83
	C3 [a]	4.96	13.73
N1s	N1 [b]	-	13.11
	N2 [b]	17.84	39.42
	N3 [b]	73.57	28.60
	N4 [b]	10.66	18.25
O1s	O1 [c]	76.22	37.55
	O2 [c]	-	34.85
	O3 [c]	23.88	27.12

[a] C1, C2, C3: C - C, C - O, and C = O bonds, respectively.
[b] N1, N2, N3, N4: pyridinic, amine, pyrrolic, and graphitic nitrogen groups, respectively.
[c] O1, O2, O3: carbonyl oxygen in quinine, carbonyl oxygen in ester and anhydride, and non-carbonyl oxygen in ester and anhydride, respectively.
[d] PL1 and PL2 refer to poultry litter collected from Arkansas and North Carolina production facilities, respectively.

The N1s spectra were deconvoluted into 4 component peaks: N1 (398.5 eV), N2 (399.2 to 399.7 eV), N3 (399.8 to 400.5 eV), and N4 (401.5 to 402 eV). N1 indicates the presence of pyridinic group in aromatics; N2 indicates amine group; N3 indicates pyrrolic group in aromatics; N4 indicates graphitic nitrogen (Ayiania *et al.* 2020). Liu *et al.* (2022) mentioned the existence of nitrogen on poultry litter surface in the forms of pyridine, amine, and quaternary forms. These data indicate that poultry litter can serve as an excellent precursor for the synthesis of biochars decorated with pyridinic, pyrrolic, and graphitic nitrogen functionalities, which have potential applications in energy storage devices such as supercapacitors.

The deconvolution of O1s peak produced three component peaks: O1 (531.3 to 531.8 eV) for carbonyl oxygen in quinine, O2 (532.4 eV) for carbonyl oxygen in ester and anhydride, and O3 (533.1 to 533.3 eV) for non-carbonyl oxygen in ester and anhydride (Chen *et al.* 2020). The carbonyl oxygen presence resembles with C=O group observed in C1s deconvolution.

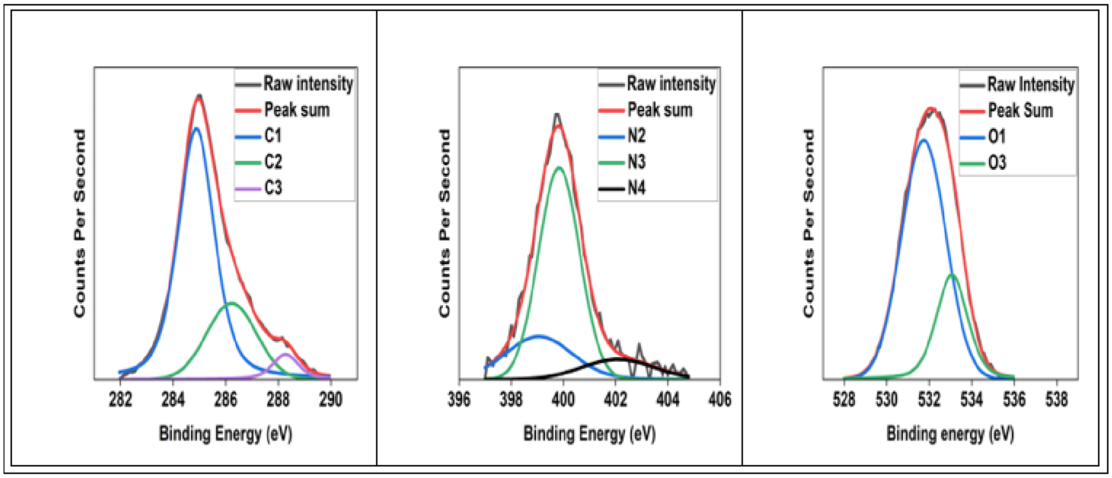

Fig. 2. XPS survey of poultry litter collected from Arkansas production facility (PL1)

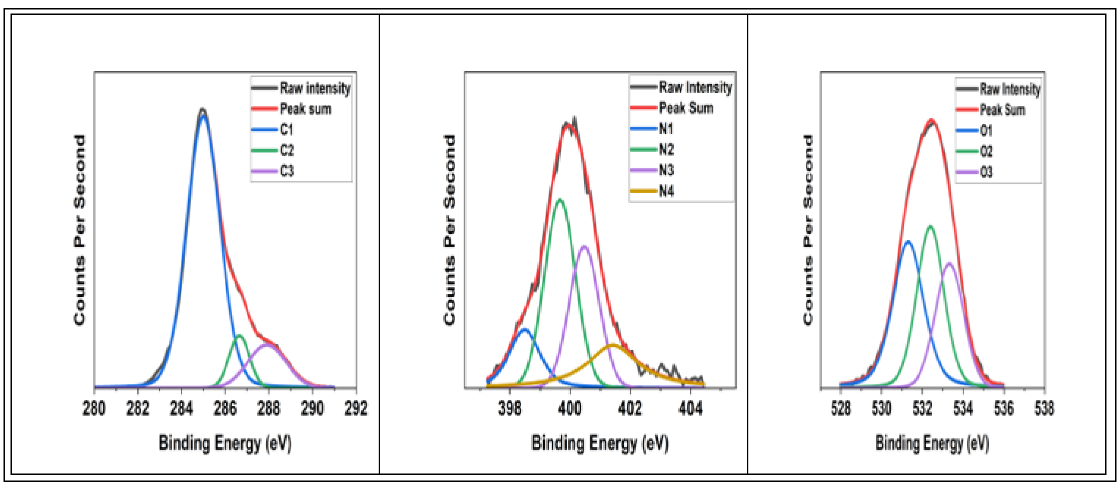

Fig. 3. XPS survey of poultry litter collected from North Carolina production facility (PL2)

ToF-SIMS analysis

The ToF-SIMS analyses of PL1 and PL2 were conducted to understand the relative distribution of ions on poultry litter surfaces. The original 2-D images of negative ion ToF-SIMS are represented in Fig. 4. A color scale used on the right side of each image describes the distributions of ions on the surface. The scale (intensity) shows the presence of ions from black and red (minimum) to dark yellow (maximum). The TC and MC in the images stand for total counts and maximum counts of ions, respectively. The negative ions observed as common between the two poultry litters are PO_2, PO_3, CN-, SO_3, CNO-, HS, and O-. The MC and TC of the ions in the two types of poultry litter are very close to each other, which indicate similar distributions of ions on the poultry litter surfaces. The contrast observed between them is the hydroxyl group absence in North Carolina poultry litter and its presence in Arkansas poultry litter. The observation partly aligns with infrared spectra, which represent hydroxyl groups presence in both poultry litters. North Carolina poultry litter did not have SO_2, whereas Arkansas poultry litter did. These variations between the poultry litter might be because of the differences in their handling and storage in two separate production facilities.

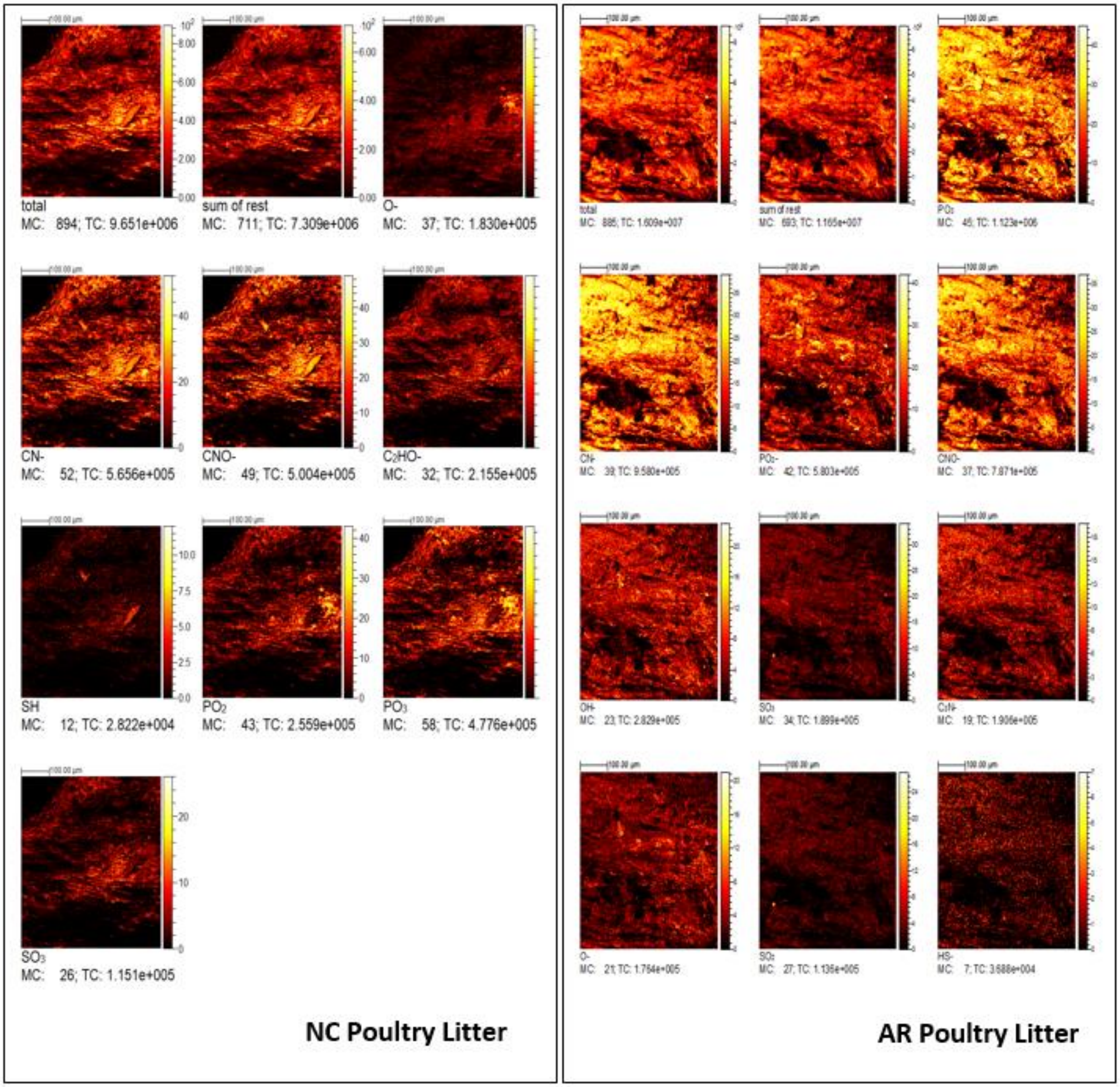

Fig. 4. ToF-SIMS original images of negative ions in North Carolina (NC) and Arkansas (AR) poultry litter

CONCLUSIONS

1. The properties of poultry litter collected from two production facilities, based in Arkansas and North Carolina were quantified and compared. Poultry litter from a production facility in Arkansas had greater fertilizer value (inorganic nitrogen, phosphorus, and potassium) than from the production facility in North Carolina; however, the latter had greater carbon content and water-holding capacity.
2. As a source of biomass energy in circular systems, poultry litter from North Carolina was better suited than from Arkansas with lower moisture content, higher bulk density, and greater C concentration likely owing to flock management and production conditions.
3. Poultry litters collected from North Carolina and Arkansas were basic in nature. The infrared spectroscopy revealed O–H, N=N, P–O, amide, SO_3H, and sulfate functional groups on the surface of both poultry litters. ToF-SIMS analyses indicated the presence of PO_2, PO_3, CN-, SO_3, CNO-, and O- ions on their surfaces. The XPS analyses confirmed the existence of carbon – carbon and carbon – oxygen bonds, amine group, and carbonyl and non-carbonyl oxygen on the poultry litter.
4. All four characterization techniques unveiled resemblances between the two poultry litters in terms of surface chemistry indicating similarity between poultry litters regardless of location perhaps because of similar feed supplied to poultry and the environment in which they were reared.
5. It is expected that the data presented in this article will provide baseline information on poultry litter and insight to researchers working with value-added poultry litter for use in circular economies.

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