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Research Article

Physio-chemical and Thermal Characterization of Demineralized Poultry Litter using Mechanical Sizing Fractioning, Acid Solvents, and Deionized Water

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Abstract

Poultry litter is an organic waste composed of manure, feathers, and bedding, containing both organic and inorganic elements. Inorganic elements corrode and agglomerate the reactors when poultry litter is thermally converted to biochar, bio-oil or combusted. Demineralizing poultry litter through mechanical size fractioning, acid solvent, and deionized water can decrease the inorganic elements' composition. This study investigates to identify the effects of different demineralization methods on the physio-chemical and thermal characteristics of poultry litter. Statistical analysis (p-value of 0.001) showed that the pretreatment methods reduced the inorganic elements. Mechanically fractioned poultry litter with particle sizes of 1.19-2.36mm and 2.37-5.00mm showed a low inorganic element composition of 7.85% and 9.19% when demineralized. The acid solvent and PL:DI treatments varied their extraction effectiveness depending on the type of inorganic element removed, ranging from 9.65-11.73% of the total inorganic element composition. PL:AcOH treatment had the lowest ash content at 6.89% from 12.33%. Mechanical size fractioned samples had the lowest moisture content, ranging from 2.53% to 4.65%. All the samples had high fixed carbon content except for PL:<1.18mm sample. Volatile matter for PL: AcOH, PL: DI, and PL:2.37-5.00mm samples were higher than the UT: PL and other treated samples. The treated samples noted a low C, high O, S, N, H/C, and O/C and comparable H content with UT: PL sample. Removal of inorganic elements increased the sample's high heating value, with the PL:2.37-5.00mm sample having the highest at 17.60 MJ Kg^{-1}. The FTIR spectra of the demineralized sample revealed a reduction in the transmittance band, indicating the decomposition of the cellulose and hemicellulose structures. TGA-DTG supported the findings by noting a shift in temperature increase with peak temperatures higher than UT: PL sample samples. The degradation occurred at temperatures up to 380°C, noting the degradation of the cellulose and hemicellulose structure of the demineralized poultry litter.

Keywords: Characterization, demineralization, inorganic elements, poultry litter

1. Introduction

Poultry litter is a renewable biomass material of chicken manure, feathers, and bedding produced from chicken production [1]. The poultry litter biomass offers a potential alternative fuel resource that can produce downstream products (biochar and bio-oil) with low nitrogen and sulfur constituents than fossil fuels, making it a promising feedstock to reduce global warming [2]. The poultry litter elemental composition depends on the type of poultry producing the biomass, operating conditions (water quality, feed type, bedding material), harvesting method, and time. The poultry litter biomass contains organic (C, H, N, and O) and inorganic elements. Alkali metals (Na, K), alkaline earth metals (Mg, Ca), transition metals (Zn, Mn, Co, Cr, Cd, Ni, Cu, Fe), post-transition metals (Al, Pb), non-metals (P, S) are the inorganic elements [2,3]. Ba, Mo, Se, Sn, U, V, Si, Cl, Ti, Cl, Na, Mn, and Ti inorganic trace metals have been reported in poultry litter from various peer-reviewed data. These inorganic trace elements are present in the biomass as salts (e.g., NaCl), free ions, or dissolved in the fluid matter inside the biomass material [3, 4].

During the thermal conversion process, the inorganic metals contribute significantly to erosion, corrosion, slagging, sintering, and equipment breakdown [5,6]. For example, a biomass reactor would require more maintenance to remove slag than a fossil reactor [5]. In addition, when disposed improperly on the ground, soil can attain high concentration levels of inorganic elements, poisoning the water bodies [7]. Under thermal process at temperatures between 200-400°C, the inherited inorganic elements evaporate, melt and retain their concentration in the biochar and bio-oil products, while above 600°C, they are retained in the ash fraction [6, 8].

Reducing inherited inorganic elements in the poultry litter is essential to improve the quality of thermal downstream products (biochar and bio-oil) without altering their properties. Also, this ensures that the biomass and its value-added products do not emit toxic pollutants to the environment and reduces the effect of damage to the thermal reactors. Mechanical size fractioning and leaching (distilled water and acid treatments) are the methods that are used to reduce the inorganic elements in biomass. Mechanical size fractioning effectively separates the ash content in the biomass according to the particle size [9]. The water leaching process is a low-cost, efficient, and easy-to-use method to remove the inorganic elements in biomass and improve the ash quality of the thermally converted biochar [10,11]. However, its effectiveness depends on the type of material, the particle size of biomass, water to biomass ratio, water temperature, and the treatment time. For example, Dayton et al. [12] demineralized straw biomass and achieved a 45 % removal rate of the total ash, which constituted K, Cl, S, Na, P, and Mg elements at an extraction efficiency of 90%, 98%, 55%, 68%, 72%, and 68% respectively in comparison to the untreated biomass [12]. A multi-step washing method can further improve the removal efficiency of the inorganic trace metals in biomass. Under moderate pyrolysis temperatures, potassium is retained in the biochar and later removed by washing with water, which also removes chlorine ions. In the multi-step washing process, potassium's removal efficiency depends on the char's properties and water temperature [13]. Acid leaching treatment effectively removes inorganic elements that are difficult to remove by water treatment [2]. However, applying high concentrations of chemicals during treatment can pollute the environment. It also alters the properties of biomass and its value-added products (biochar and bio-oil) [4].

Much research has concentrated on the efficiency of the demineralization process of poultry litter using mechanical fractioning and leaching agents, but few systematic reports focus on comparing the demineralized physio-chemical structure and thermal behavior. Therefore, the study aims to investigate how different demineralization processes affect the physio-chemical structure of the demineralized poultry litter derived from broiler chickens. In addition, a null hypothesis stating that pretreatment methods do not reduce inorganic elements was analyzed.

2. Materials and Methods

2.1 Feedstock preparation

Poultry litter constituting a bedding material of sunflower husks, wood shavings, and manure was collected from the Tshipane poultry farm in Palapye, Botswana, and used in this study. The feedstock was dried in an oven (Systronix Scientific, 278, South Africa) at 105°C for 24h, pulverized using a laboratory ball mill (Pulveisette 6, FRITSCH, Germany) for 15mins, and sieved into the following sizes: <1.18mm, 1.19-2.36mm and 2.37-5.00mm corresponding to the available mesh sizes.

2.2 Demineralization process

Mechanical size fractioning and leaching treatment processes were adopted in this study to remove the inorganic elements from the poultry litter. Mechanical size fractions of the biomass were categorized into the following size groups: <1.18mm, 1.19-2.36mm, and 2.37-5.00mm and labeled as PL:<1.18mm, PL:1.19-2.36mm, and PL:2.37-5.00mm respectively. The study adopted poultry litter particle size between 1.00-2.00mm for the solvent leaching process, as Rodríguez-Machín et al. [14] reported that this size reduces the mass transfer limitations during the leaching process, making it effective on biomass to solvent interaction process. Five solvents were used in the experiment and are as follows: acetic acid (AcOH), sulfuric acid (H_2SO_4), hydrochloric acid (HCl), phosphoric acid (H_3PO_4), and deionized water (DI) with samples labeled as PL:AcOH, PL:H_2SO_4, PL:HCl, PL:H_3PO_4 and PL:DI respectively. The acidic solvents were diluted at 5% solution with all solvents to biomass ratio of 10:1 (v/w) reacting at room temperature (25 ± 3°C) for 2h while being stirred continuously with a magnetic stirrer [1]. The biomass samples were then rinsed with deionized water until a pH (Envtek, ENV-49, India) of 7 was attained. They were subsequently dried in an oven at 105°C for 24h, and then sealed in a desiccator for storage [1]. The demineralization extraction efficiency was determined using Equation 1.

$$\frac{M_{UT}-M_{T}}{M_{UT}}\times 100\% \qquad (1)$$

Where: M_{UT} is the mass of untreated poultry litter, and M_T is the mass of the demineralized poultry litter.

2.3 Characterization

Samples under study were prepared for analysis by pulverizing (Pulveisette 6, FRITSCH, Germany) into 125µm particle size. All the experiments were conducted in three replicates to determine the mean and the standard deviation. Inorganic trace elements, proximate, ultimate, and Fourier-transform infrared (FTIR) analysis were the physio-chemical characterizations conducted in this study. The thermal characterizations under study were higher heating values and thermogravimetric analyses.

2.3.1 Inorganic trace elements analysis

The inorganic trace elements in the samples were determined using a handheld portable X-ray fluorescence machine (Olympus Delta-50 Premium, USA). The instrument has a Ta anode, a maximum output of 4W. Prior to use and after 20 scans the instrument is calibrated using a 316-alloy stainless steel coin configured to geochemist mode (50 kV and 10 kV beam configuration) set at 30 s per each beam, making a total of 60 s per each complete scan. The device had silicon drift detectors for measuring fluorescence and rhodium tubes from which X-rays were released. A sample with a thickness of 2 cm or more was placed beneath the instrument aperture to ensure proper penetration. Also, the instrument was repositioned on the same sample after every scan to determine the material's intrinsic heterogeneity.

2.3.2 Proximate analysis

The thermogravimetric analyzer (Leco TGA 701, USA) determined the sample's moisture, ash, volatiles, and fixed carbon. The ASTM D7582 MVA in biomass was used as a standard method to measure the sample's moisture, ash, and volatile content. Empty crucibles were loaded into the furnace carousel to zero weight, and samples of 1.00-2.00g were then loaded into the crucible. Moisture content was determined by heating the samples at 15°Cmin^{-1} to 105°C in a nitrogen atmosphere (7Lmin^{-1}). The volatile content was determined by further heating the samples at 50°Cmin^{-1} to 700°C in a nitrogen atmosphere. The ash was obtained by heating the samples at 50 °Cmin^{-1} to 750°C in an oxygen atmosphere till constant weight was achieved. Fixed carbon was obtained by subtracting from 100 the measured moisture, ash, and volatiles percentile values.

2.3.3 Ultimate analysis

About 1.75-1.85g of the sample was weighed using a mass balance (Fisherbrand CSC501, USA) and transferred into a universal soft tin cup. The cup was folded using tweezers and placed into an autosampler. The folded samples on the autosampler were transferred into the furnace of an elemental analyzer (Thermo Scientific Flash 2000 CHNS/O, USA) set at 950°C in the presence of oxygen gas for flash combustion. The helium gas transported the gaseous combustion products (N_2, NO_x, H_2O, SO_2, and CO_2) through the CuO column, where NO_x and O_2 were reduced to N_2 and CuO, respectively, while water was absorbed in another column. The combustion gases discharged from the CuO column were captured in the temperature-bound desorption (TPD) column, except for the N_2 gas that went through the column. A programmed temperature rise inside the column ensured the release of gases separately passing through a thermal conductivity detector (TCD), producing an electrical signal proportional to the concentration of nitrogen, carbon, hydrogen, and sulfur. The oxygen content was calculated by subtracting the sum of weight percentages of nitrogen, carbon, hydrogen, sulfur, and ash from 100%.

2.3.4 Higher heating value (HHV)

An oxygen bomb calorimeter (IKA C6000, USA) was used to determine the sample's higher heating value under the standard specification of ASTM D5865-12 [14]. Two grams of sample were weighed using a mass balance (Adam PGW438, USA), transferred into a ceramic crucible with the samples connected to the ignition wire using a cotton thread, and placed in a thermally insulated vessel containing a temperature transducer that records temperature change during the combustion process. The vessel was closed, filled with 30bar pressure of oxygen, and combusted. The heat generated is the higher heating value of the samples under study.

2.3.5 Fourier-transform infrared (FTIR) analysis

Approximately 50mg of each sample was subjected to a Fourier-transform infrared (FTIR) analysis using an FTIR spectrometer (Bruker Vertex 70v, USA). The spectra were obtained at a resolution of 4 cm^{-1} at a scan rate of 16$cmmin^{-1}$ in the 4000-400cm^{-1}. The sample was placed onto a total reflectance cell (ATR), and a scan was run to generate a spectrum in which the OPUSTM software identified its functional groups. Prior to each analysis, the ATR cell was cleaned with ethanol.

2.3.6 Thermogravimetric and differential thermogravimetric analysis (TGA and DTG)

The thermal degradation performance of the samples understudy was evaluated using a thermogravimetric differential machine (Mettler Toledo DSC/TGA 3+, USA). The samples (approximately 10mg) were placed into the crucibles, weighed using a high ultra-precision mass balance attached to the thermogravimetric analyzer, and placed in the furnace chamber. The N_2 gas flowing at 50mlL^{-1} with a heating rate of 10°$Cmin^{-1}$ and a temperature range of 25°C to 915°C were the operating parameters adopted [13]. The weight loss was converted to percentiles to ensure consistency and uniform comparison.

2.4 Statistical analysis

The effect of pretreatment methods on the inorganic elements in the poultry litter was statistically analyzed. A null hypothesis states that the pretreatment methods do not reduce the inorganic elements. The alternative hypothesis states that the pretreatment methods reduce the inorganic elements. A descriptive statistic with one-way ANOVA at 95% confidence interval was used to test the null hypothesis, a p-value of less than 0.05 will reject the null hypothesis and accept the alternative hypothesis. R-software (version 4.3.2) was used for the analysis.

3. Results and discussion

3.1 Inorganic elemental analysis

Inorganic elements composition and demineralization efficiency of treated and untreated poultry litter are listed in Figure 1 and Table 1, respectively. The statistical analysis test showed an F-value of 26.02 and p-value of 0.001 concluding that they are 0.1% chance that the F-value might occur due to noise hence rejecting the null hypothesis and accepting the alternative hypothesis which states that the pretreatment methods reduce the inorganic elements. Twelve inorganic elements were identified and categorized as non-metals (Si, P, S, and Cl), inorganic metals (K, Ca, Al, and Fe), and trace metals (Cr, Mn, Cu, and Zn). All the samples except for the PL:HCl sample had inorganic Cl composition that was less than 1.00 %. Inorganic Cl in the biomass has to be less than 0.97 % to ensure an insignificant reaction with any metal during thermal conversion, reducing the possibility of corrosion of the thermal reactors [15–17]. However, PL:HCL sample had a high Cl composition of 5.09% above the desired level due to the presence of Cl molecules in the HCl solvent, making the feedstock undesirable to expose to high temperatures during the thermal process. The extraction efficiency of Cl was high in the PL:H_2SO_4, PL:1.19-2.36mm samples at 100.00% and 72.67%, respectively (Table 1). The findings corroborated with results obtained by other researchers who noted the effectiveness of mechanical fractioning and chemical solvents with non-Cl solvent molecules in demineralizing the inorganic Cl composed in the biomass [18–20].

Inorganic P was reduced in all sample treatments except in the PL:H_3PO_4 sample. The inorganic P was less than 1% on all samples except for the PL:H_3PO_4 sample which had 3.86% with an additional percentage of 423.11% (Table 1) in comparison to the untreated poultry litter (UT:PL). Our findings agreed with other researchers who noted a significant reduction in the inorganic P after treatment with acids that do not constitute P [13,21]. High inorganic P in biomass results in high ash production when combusted, which results in fouling, slagging, emissions of volatiles, and corrosion of the combustion systems [22]. However, the presence of inorganic P in the biomass acts as a catalyst when pyrolyzed, thus favoring the production of char and gas over tar and improving the char-to-carbon burnout when combusted [23].

Hence the demineralized PL:H_3PO_4 sample will favor char production and produce quality char with good burnout compared to other treated samples. All the treatment methods except for PL:H_2SO_4 reduced the inorganic S effectively, with the highest at 45.70% (Table 1) of the PL:1.19-2.36mm sample. H_2SO_4 added the inorganic S at a factor of 652.35% (Table 1) compared to UT:PL. Wigley et al. [21] noted a significant reduction of the inorganic S when the biomass was demineralized with the acids in the study [21].

To ensure a non-reaction of inorganic S with active metal oxides to form metal sulfides during combustion, which results in bed agglomeration, the S composition should be less than 0.20% [24]. However, all the demineralized samples and the untreated poultry litter have inorganic S composition greater than 0.20 %; hence a need to install desulphurizer units on combustion units is of relevance [15].

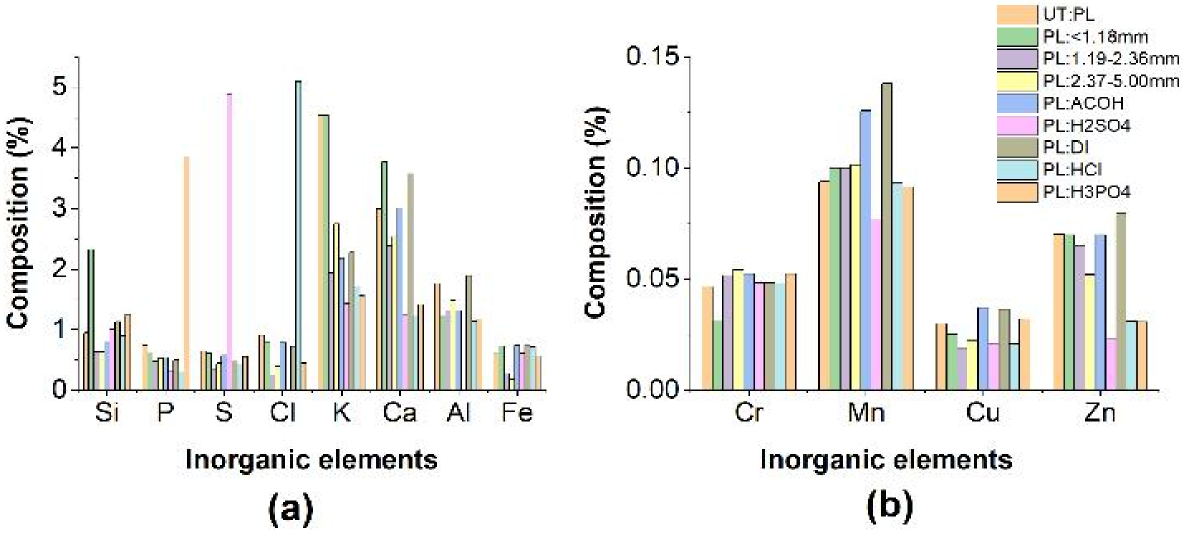

Figure 1. Inorganic elements of untreated and treated poultry litter (a) Elements (b) Trace metals.

Table 1. Demineralization extraction efficiencies of poultry litter under different treatment methods.

Elements	PL:<1.18mm	PL:1.18-2.36mm	PL:2.37-5.00mm	PL:AcOH	PL:H_2SO_4	PL:DI	PL:HCl	PL:H_3PO_4
Si (%)	-144.21	33.68	33.68	14.74	-6.32	-18.95	5.26	-31.58
P (%)	18.69	36.17	27.59	27.06	56.31	32.01	58.37	-423.11
S (%)	6.47	45.70	31.57	11.28	-652.35	25.49	32.54	13.32
Cl (%)	13.11	72.67	56.72	13.22	100.00	20.87	-460.65	50.26
K (%)	0.06	57.37	39.45	52.03	68.57	49.91	62.31	65.52
Ca (%)	-25.56	20.31	15.53	-0.17	58.65	-18.97	58.92	52.67
Al (%)	30.11	25.57	15.34	25.57	100.00	-7.39	35.23	32.95
Fe (%)	-21.92	53.31	69.36	-23.83	-1.69	-25.09	-19.25	5.14
Cr (%)	37.20	-3.60	-8.40	-4.40	3.20	3.00	3.80	-4.40
Mn (%)	-11.11	-11.11	-12.67	-39.67	14.33	-53.33	-3.44	-1.67
Cu (%)	15.67	38.00	26.00	-23.67	29.33	-21.33	30.00	-7.00
Zn (%)	0.29	7.29	25.57	0.14	66.57	-13.43	55.71	55.86

Mechanical size fractioning (PL:1.19-2.36mm, PL:2.37-5.00mm) and acid (PL:AcOH and HCl) samples reduced the inorganic Si. PL:<1.18mm, and the other samples attained more Si composition in their biomass. The inorganic Si composition in the treated samples shows that the untreated poultry litter was contaminated with soil before demineralization. Si element has a good bond strength with the biomass matrix [25]. The inorganic Si in the mechanical size fractioning (PL:1.19-2.36mm, PL:2.37-5.00mm) samples had a weak bond strength with the biomass matrix; hence upon fractioning, they were reduced effectively and accumulated in the PL:<1.18mm sample. For the solvent treatment, the AcOH and HCl acids dissolved the Si in the biomass matrix, reducing the composition. Another possible increase in Si composition in PL:H_2SO_4 and PL:H_3PO_4 samples might be due to the use of laboratory glassware during experiments; hence they degrade when exposed to acids [25,26]. The findings were in agreement with other researchers [13,18,21,27], who noted an increase in the inorganic elements when treated with acid solvents. ,

Acid treatment and 1.19-2.36mm size fractioning effectively reduced the inorganic K compared to DI, 2.37-5.00mm, and <1.18mm size fractioning methods. Rodríguez-Machín et al. [25] noted a high removal fraction of inorganic K when the biomass was leached with acid solutions (weak organic acid, HCl, and H_2SO_4), which agrees with our results shown in Figure 1. The inorganic K in biomass exists as a K^+ ion, making them highly soluble in weak acid (AcOH) and DI solvent treatments. The PL:HCl, PL:H_2SO_4, PL:H_3PO_4, PL:1.19-2.36mm, and PL:2.37-5.00mm methods effectively removed Ca inorganic metal at extraction efficiency of 58.90%, 58.70%, 52.70%, 20.30%, and 15.50% respectively. However, PL:<1.18mm, PL:DI, and PL:AcOH methods attained undesirable Ca content in the treated poultry litter, with PL:<1.18mm method having the highest fraction compared to PL:DI and PL:AcOH methods. Inorganic Al was removed entirely in the PL:H_2SO_4, reduced significantly in the mechanical size fractioning and acid treatment methods. Only the PL:DI sample added the Al composition at a factor of 7.39% compared to the UT:PL sample. The inorganic Fe was significantly reduced in the PL:1.19-2.36mm, PL:2.37-5.00mm, and PL:H_3PO_4 samples, while the rest of the treatment methods retained the metal. The high composition of inorganic Al and Fe in the untreated poultry litter is inherited from the poultry feed [28]. The removal of Al is high in acids compared to DI and mechanical size fraction. The latter observation may be attributed to the nature of the strong acid that accelerates the reaction time to remove the inorganic Al [25]. The inorganic Fe exists in ferric form (Fe^{3+}), which is insoluble in solvents, and when treated, they prove ineffective compared to mechanical size fraction at particle size above 1.19 mm [25]. The findings were in agreement with other research [1,2,13,29], which noted an effective reduction of inorganic metals with acid solvents and mechanical size fractioning.

The inorganic trace metals (Cr, Mn, Cu, and Zn) had an insignificant composition of less than 0.10% on all the demineralized samples. The Cr element in PL:<1.18mm, PL:HCl, PL:DI, and PL:H_2SO_4 samples was reduced, while Mn element composition was retained in all the treatments. The mechanical size fractioning and strong acids (HCl and H_2SO_4) reduced the Cu composition. The Zn composition was reduced in all the treatments except in the PL:DI sample, PL:<1.18mm, and PL:AcOH samples.

Overall, the inorganic elements in the poultry litter were extracted at different efficiencies depending on the type of demineralization process. After treatment, some demineralized poultry litter increased their inorganic composition caused by inorganic elements in the treatment solvents that dissolve and add to the constituent of the treated biomass. However, the total inorganic element composition was reduced regardless of the solvent applied. Other researchers [11,13] observed an increase in inorganic element composition after treatment of the respective biomass, which they noted resulted from the presence of inorganic elements in the treatment solvents.

3.2 Proximate, ultimate analysis, and higher heating value

Table 2 shows untreated and treated poultry litter's proximate, ultimate analysis, and high heating value. Compared to the UT:PL, the ash content of the following treated samples: PL:AcOH, PL:2.37-5.00mm, PL:DI, PL:HCl, PL:H_2SO_4, and PL:1.19-2.36mm decreased by 5.44%, 4.91%, 4.17%, 2.83%, 2.69% and 0.92% respectively from 12.33% to 11.44-6.89%. However, the PL:H_3PO_4 and PL:<1.18mm samples increased their ash content by 1.17% and 25.62%, respectively, from 12.33% to 13.50% and 37.95%, respectively. This increase in ash content is due to the recalcitrant of P (423.11%) and Si (31.58%) in the H_3PO_4 solvent of the PL:H_3PO_4 sample and retained high fraction of Si (114.21%) and Ca (25.56%) on the 1.88mm poultry litter size when fractioned to PL:1.18mm sample (Table 1). The findings were similar to other researchers [1,13] who noted the presence of Mg, Ca, Si, Al, etc., in the samples results from an increase of ash content as these inorganic elements are insoluble in the demineralization solvents or retained at high concentration on small particle size fractioning. After deionized water treatment, the PL:DL sample lost 4.91% of the inorganic elements in the ash by dissolving in water. The acid treatments except for PL:H_3PO_4 lost ash content ranging from 2.83 to 5.44%, which dissolved in the acid solvents that is a water and acid mixture. Hence the inorganic elements composed in the acid treatments are mainly soluble in water compared to the acids, and they exist as water-soluble components mainly in the form of Cl^-, NO_3, CO_3, and PO_4^{3-} [30]. The PL:DI and PL:2.37-5.00mm samples can significantly reduce the ash content in the poultry litter, thus reducing the possibility of agglomeration, slagging, corrosion, and deposition in the thermal conversion processes [27].

All the demineralized samples had low moisture content compared to the UT:PL. The mechanical size fractioned samples had the lowest moisture content ranging from 2.53% to 4.65%, compared to the acid treatment and the PL:DI samples which ranged from 4.26% to 5.75%. The PL:DI moisture samples were comparable with the acid treatment samples. This is because all of the biomass samples absorb water molecules during treatment and only a part of the moisture evaporates during oven drying. The sample with the smallest particle size (PL:<1.18mm) had the lowest moisture content compared to the other mechanical size fractioned samples. The findings were in agreement with Klinger et al. [31], who noted an increase in moisture content when particle size of corn stover biomass increased. High moisture results in more energy required for thermal conversion and affects the quality of pyrolysis oils (phenol, toluene, and benzene) [32].

All the treated samples except for PL:<1.18mm sample increased their fixed carbon with acid treatments (PL:H_3PO_4 > PL:HCl > PL:H_2SO_4 > PL:AcOH) being the highest followed by mechanical size fraction sample (PL:2.37-5.00mm > PL:1.19-2.36mm) while PL:DI sample is comparable to PL:1.18-2.36mm samples. The volatile matter for PL:AcOH, PL:DI, and PL:2.37-5.00mm samples were high compared to the UT:PL samples.

However, the other samples had a low volatile matter, with PL:<1.18mm sample being the least, while others were comparable to the UT:PL sample. A low volatile matter shows that most of the hemicellulose in the biomass is devolatilized during thermal conversion at temperatures below 275.00°C compared to cellulose and lignin [33]. The demineralization process improves the poultry litter's fuel properties, enabling it to ignite and combust better while preventing high-temperature corrosion, agglomeration, and fouling in the thermal reactors [32]. The effects of the demineralization process on the poultry litter elemental composition were evaluated using ultimate analysis, and results are presented in Table 2. In comparison with the UT:PL sample, the treated samples had low C, high O, S, N, H/C, and O/C and with comparable H composition.

PL:<1.18mm and PL:1.19-2.36mm had the lowest and highest C content at 19.63% and 41.24%, respectively, compared with the other demineralized samples. Our findings were in agreement with Javed et al. [34] and Kasim et al. [35], who noted a reduction of C composition on demineralized biomass [34,35]. Mechanical fractioning reduces the crystallinity of the cellulose, while solvent demineralization eliminates the lignin and degrades the cellulose hence causing a reduction in the C composition [36,37]. Other researchers [33,34] reported a decrease in the O composition of the demineralized biomass compared to the untreated biomass, and they noted that the cause was the acid solvents used in the treatments, which resulted in deoxygenation of the biomass.

The difference between our findings is in the biomass type used; the treated poultry litter tends to absorb the oxygen molecules through treatment solvents, and when fractioned, they accumulate the oxygen long chain polysaccharides cleavage on mechanical fraction. High O in poultry litter promotes moisture absorption, decreasing shelf life [33]. The H composition was insignificant with the UT:PL sample, and the slight increase might be due to the removal of soluble inorganic elements that increments lignocellulosic content hence adding the H content [18].

Table 2. Proximate, ultimate, and high heating value analysis of untreated and treated poultry litter.

Samples	Proximate analysis (%, air-dry basis)				Ultimate analysis (%, air-dry basis)							HHV (MJ/kg)
	M/C	VM	Ash	FC	N	C	H	S	O	H/C	O/C	
UT:PL	9.74	70.07	12.33	17.61	0.00	44.73	5.22	0.00	41.30	0.117	0.92	14.89
PL:< 1.18mm	2.53	50.18	37.95	11.87	1.37	19.63	2.65	1.15	66.45	0.131	1.04	8.14
PL:1.19-2.36mm	3.54	69.45	11.41	19.14	2.24	41.24	5.42	1.20	58.85	0.135	3.39	16.71
PL:2.37-5.00mm	4.65	71.86	7.42	20.72	2.20	37.50	4.92	1.18	45.45	0.131	1.43	17.60
PL:AcOH	5.73	73.03	6.89	20.07	2.46	37.20	4.95	1.19	45.45	0.131	1.21	15.90
PL:H_2SO_4	5.44	65.58	9.64	24.78	2.30	33.83	4.24	0.00	59.64	0.133	1.22	15.75
PL:DI	5.37	72.10	8.16	19.74	1.72	40.70	5.35	1.23	42.25	0.125	1.76	15.65
PL:HCl	4.93	64.57	9.50	25.93	2.25	39.40	5.34	1.26	43.00	0.136	1.09	15.14
PL:H_3PO_4	4.26	60.38	13.50	26.12	1.94	37.48	5.05	1.16	45.62	0.135	1.22	16.43

The removal of soluble inorganic elements and the presence of N and S in the solvent treatments resulted in the addition of these constituents to the demineralized poultry litter samples [18]. N and S elements in poultry litter are found in small quantities, less than 2.50%; however, when combusted, they release NO_x and SO_x into the environment, thus polluting and contributing to global warming [33]. Due to the high molecular mass of C, the H/C ratio shows variances in the amount of hydrocarbons contained in the poultry litter [1,27]. The H/C ratio of the demineralized poultry litter increased after treatment compared to the UT:PL sample. This increase in the H/C ratio might be due to the removal of organic matter (reduction in C composition), thus reducing carbonaceous matter [27]. Javed et al. [34] noted a decrease in the H/C ratio of the treated biomass with AcOH compared to the untreated biomass [34]. Those findings differ from ours because solvents act differently on different biomass. The O/C ratio of demineralized poultry litter was higher than UT:PL. This difference is due to the C composition between the treated and untreated poultry litter [32,34]. The demineralized poultry litter samples, except for PL:<1.18mm, had higher heating values greater than that of the UT:PL sample (Table 2). The higher heating value is inversely proportional to the ash content; hence the decrease in ash content is the increase in the higher heating value [33]. The ash content for PL:<1.18mm was the highest at 37.95% compared to the other treatments and UT:PL samples ranging between 7.42-13.50%, that is why it has the lowest higher heating value of 8.14MJ/kg.

3.3 Identification of surface functional groups using FT-IR spectrometry

Qualitative analysis of the chemical structure functional group on untreated and treated poultry litter samples was determined using the FTIR spectrometer, as shown in Figure 2. The spectra of the untreated and treated poultry litter showed a significant difference in intensity, which clearly shows that the demineralization process alters the poultry litter cellulose, hemicellulose, and lignin structure to a certain extent.

In the FTIR spectrum, a wavelength at a range of 3979-3066cm^{-1} is observed on UT:PL and demineralized poultry litter samples which represents the presence of O-H stretching vibration of hydroxyl functional groups in alcoholic, carboxylic, and phenolic structures [35,38]. This signifies the presence of H_2O molecules with UT:PL sample having a high moisture content (Table 2) with low transmittance compared to other demineralized samples except for PL:<1.18mm and PL:H_2SO_4 samples. The effect of the demineralization process on the O-H band transmittance and the UT:PL is negligible on each other, with moisture content present in all samples being less than 10%. However, removing the O-H group through evaporation improves the hydrophobic behavior of all demineralized samples except for PL:<1.18mm and PL:H_2SO_4 samples in comparison to the U:PL sample [33].

A spectrum wavelength of 3000-2680cm^{-1} is observed on all samples, with differences in their absorption transmittance intensity. This signifies the presence of asymmetric C-H stretching vibrations in aliphatic compounds of CH_2 and CH_3 groups of the alkanes and alkenes in the samples, respectively [34]. The transmittance of these samples PL:DI, PL:H_3PO_4, PL:2.37-5.00mm, PL:HCl, and PL:1.19-2.36mm were high compared to UT:PL sample, which signified the decomposition of the hemicellulose structure of the carbonyl groups, decarboxylation, and glycosidic bond breakage due to the demineralization process [35]. PL:<1.18mm, PL:AcOH and PL:H_2SO_4 samples, compared to UT:PL sample, appreciated the asymmetric C-H stretching strengthening the carbonyl functional group due to the decrease in their transmittance being observed to the lowest.

All the samples showed absorption at a wavelength range of 2079-1567cm^{-1}. The spectra range of 1650-1510 cm^{-1} shows the vibration of C=C stretching in the aromatic compounds present in the lignin structure due to carbonate impurities [34]. The intensity of the demineralized samples, except for PL:AcOH declined due to the high removal of impurities (carbonates) [39]. The PL:AcOH is leached with an organic less acid solution (AcOH) which promotes the formation of carbonates on the poultry litter, resulting in a more intensity band compared to other samples [39]. A wave number 2100cm^{-1} shows the presence of C≡C bond present in all the samples of lignin [35]. All the demineralized samples showed an increase in intensity which is low transmittance, with the PL:<1.18mm sample having the highest intensity. This is due to the splitting of cellulose and hemicellulose in the poultry litter due to the dihydroxylation, decarboxylation, and hydrolysis reactions [33]. The increase in the intensity of the alkyne bands shows a reduction in cellulose and hemicellulose content and an increase in lignin content [33]. At 1810-1771cm^{-1}, C=O stretching comes from the free carbonyl group in the poultry litter hemicellulose [25]. The PL:AcOH sample has the most disrupted carbonyl and phenyl groups compared to other samples, with the significance of other treatments showing a reduction in the specified group [33].

The broadband 1490-1280cm^{-1} was identified in all samples, with UT:PL sample having the highest intensity compared to the demineralized samples. This wavelength range signifies the presence of aromatic amines (C-N), O-H, C-H, and C-O stretching vibrations through the dehydration and decarboxylation of hemicellulose, cellulose, and lignin, which improves the carbon and higher heating value of the poultry litter samples (Table 2). The broadband 1186-839cm^{-1} signified the presence of -OH, P^+-O^-, and C-O in the cellulose of the poultry litter samples. UT:PL sample has the most intensity compared to the demineralized samples due to the cross-linking of the C-OH, C-O-C, and other functional groups in the demineralized samples [34].

3.4 Thermogravimetric and differential thermogravimetric analysis (TGA and DTG)

The effect of inorganic element removal from the poultry litter by the demineralization process was analyzed by studying the weight degradation with respect to temperature [40]. The thermogravimetric (TG) and derivative thermogravimetric (DTG) profiles of untreated poultry litter and demineralized poultry litter are shown in Figure 3a-b, respectively. As shown in Fig.3a, all the samples underwent three degradation stages: dehydration, devolatilization, and carbonation. All samples were dehydrated at a temperature range between 33.00-142.00°C. Kasim et al. [35] noted moisture removal on the biomass surface at 129.00°C, which is within the temperature range in our findings.

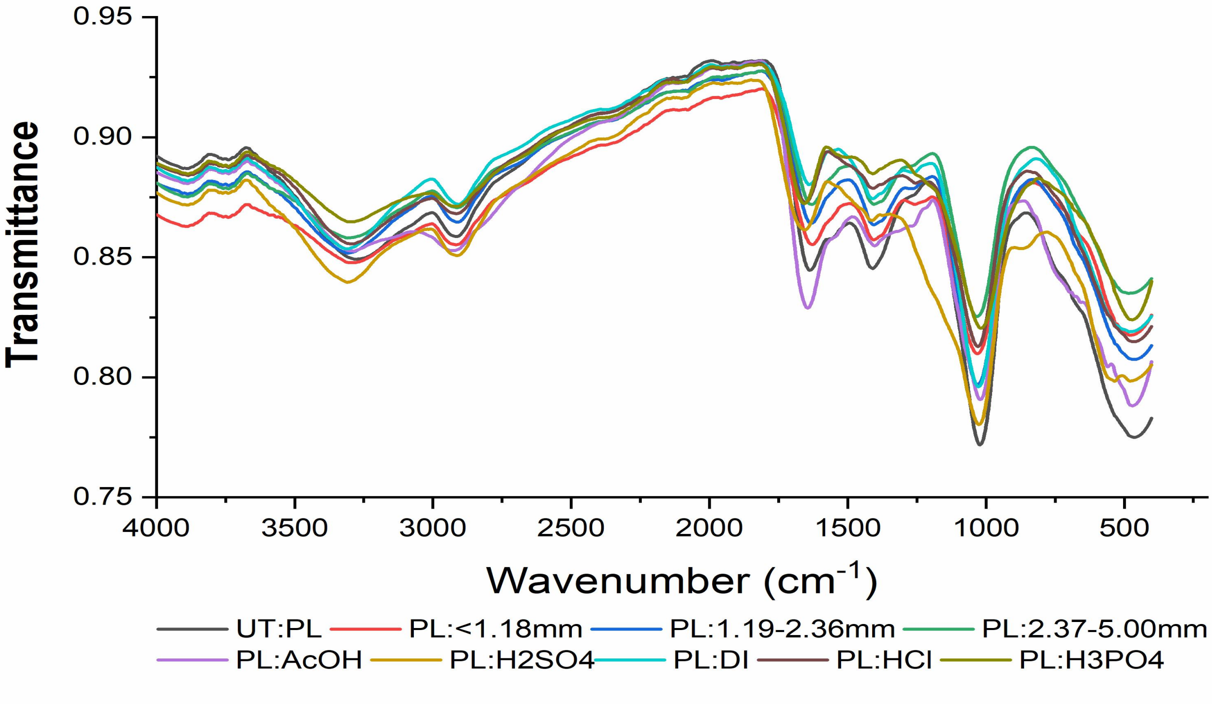

Figure 2. FTIR Spectra of untreated and treated poultry litter.

The devolatilization process of all the samples occurred at a temperature range between 200.00-380.00°C (Figure 3b). This stage signifies the hemicellulose and cellulose structure degradation in poultry litter [33]. Compared to the UT:PL sample, all treated samples shifted their maximum peaks to high temperatures (Table 3), indicating a high temperature required to decompose the hemicellulose and cellulose structure. This shift can be a possible result of the high active pyrolysis of hemicellulose and cellulose in the UT:PL sample due to the presence of high inorganic elements compared to the demineralized samples. Hence the active pyrolysis of UT:PL starts at a lower temperature compared to the demineralized samples [34]. Our findings were supported by other researchers [33,34] who justified the effect of inorganic elements in shifting the maximum peak temperatures compared to the untreated biomass. During the devolatilization, two peaks were observed on all samples except on PL:H_3PO_4 and PL:<1.18mm. Two peaks separate the degradation of hemicellulose and cellulose, while a single peak overlaps these structures [34].

In comparison to the UT:PL, PL:HCl, PL:<1.18mm, and PL:H_2SO_4 devolatilized at low temperatures 186.00°C,

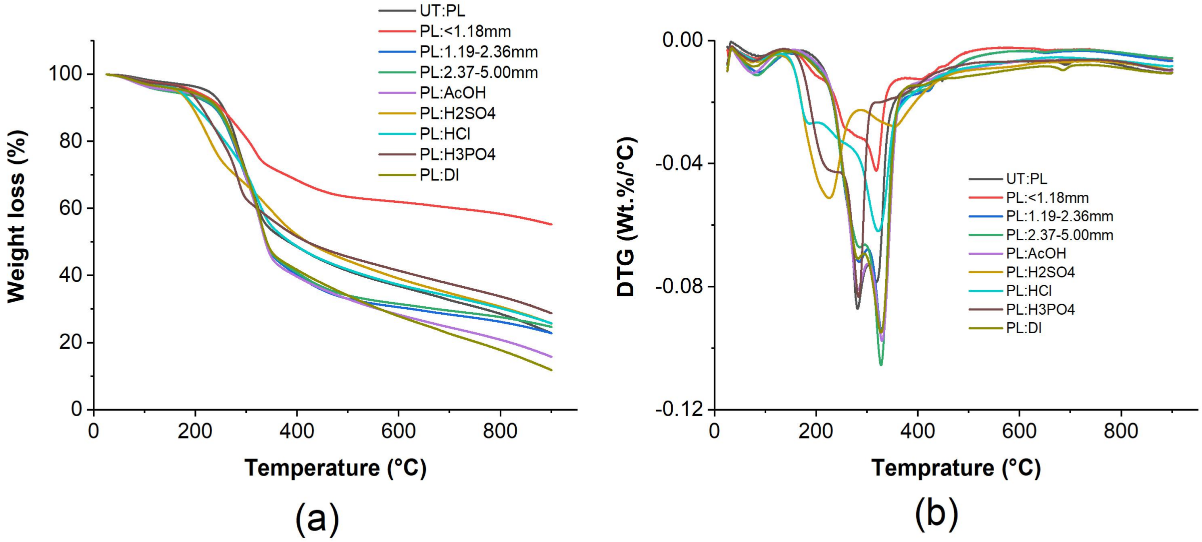

Figure 3. Untreated and treated poultry litter (a) TGA (b) DTG.

Table 3. Thermogravimetric analysis for untreated and treated poultry litter.

Sample name	Peak Temperature (°C)		Derivative mass loss (%/°C)	
	1	2	1	2
UT:PL	281.00	318.17	-0.09	-0.08
PL:<1.18mm	201.83	317.50	-0.01	-0.04
PL:2.36-1.18mm	284.00	328.67	-0.07	-0.09
PL:5.00-2.37mm	285.18	327.17	-0.07	-0.11
PL:AcOH	281.30	329.33	-0.08	-0.10
PL:H2SO4	225.17	353.83	-0.05	-0.03
PL:DI	282.17	327.17	-0.07	-0.09
PL:HCl	186.67	321.67	-0.03	-0.06
PL:H_3PO_4	283.00	-	-0.08	-

201.00°C, and 225.00°C. This is because the PL:HCl, PL:<1.18mm, and PL:H_2SO_4 samples contain a high composition of Cl, Si, and S elements, respectively which contributes to the active formation of free radicals through homolytic cleavage covalent bonds lowering the active pyrolysis temperature that degrades the hemicellulose structure [35]. Other researchers [1,4] were in agreement with our findings. PL:2.37-5.00mm, PL:AcOH, PL:H_2SO_4, PL:DI, and PL:HCl samples due to low ash content of less than 10.00 % (Table 2) tend to be more thermal stable with less effect on catalytic behavior [35]. In addition, the PL:2.37-5.00mm, PL:AcOH, PL:H_2SO_4, PL:DI, and PL:HCl samples have a high peak height (Table 3) compared to UT:PL, PL:HCl, PL:<1.18mm and PL:H_2SO_4 samples signifying high reactivity as reactivity is directly proportional to the peak height and inversely proportional to the peak temperature. Javed et al. [34] noted a high reactivity linked to the high peaks for wheat straw which was treated with CH_3COOH acid [34]. Lignin decomposition occurs at a wide range from 380.00-670.00°C at a slow rate with non-observable peaks on the samples, thus the carbonation process. Also, the FTIR analysis of the treated samples had different degrees of effects on the physicochemical structure, and comparing their peak temperatures, their variance is significant, indicating a structural effect change to the treated samples; hence the removal of inorganic elements played a significant role in the thermal behavior of the biomasses under study [1].

4. Conclusion

This work has studied the effects of demineralization of poultry litter using mechanical size fractioning, acid solvents, and deionized water. The demineralization process extracted the inorganic elements at different efficiencies depending on the solvent type or fractioned size. Inorganic elements in the treatment solvents resulted in additional composition to the final treated sample. All treatments except for PL:H_3PO_4 and PL:<1.18mm directly affected the ash content, which showed a direct relation to the amount of inorganic composition contained in the treated poultry litter. The reduction of inorganic elements resulted in reduced moisture content and increased fixed carbon and volatile matter. Upon elemental characterization, the demineralized poultry litter attained a high H/C and O/C ratio due to removing organic matter after treatment. These results are supported by an increased intensity of the functional group peaks observed in the FTIR spectra. According to the TGA-DTG, the demineralized sample showed an increased shift in the temperature range and peak temperature with two degradation processes (dehydration and devolatilization) of the cellulose and hemicellulose observed at temperatures below 380°C. The higher heating value of the demineralized poultry litter with low inorganic elements improved. Overall, the demineralization process improves the poultry litter's physio-chemical and thermal properties, making it the best-suited option for inorganic element removal before the thermal conversion process.

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Authors Contribution

Kevin Nyoni: Conceptualization, methodology, formal analysis, writing—original draft Leungo Kelebopile: supervision, resources, project administration

Conflicts of Interest

The writers report no conflicts of interest.

Data Availability statement

The data presented in this study are available on request from Kevin Nyoni (send email to kevin.nyoni@studentmail.biust.ac.bw).

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