Drying temperature - duration impacts on moisture, carbon, and nitrogen losses from broiler litter

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Abstract: Drying poultry litter (PL) prior to analysis minimizes nutrient transformations and losses and facilitates sample preparation. Drying also allows comparison among different litters on dry-weight (dw) basis. However, losses of carbon (C) as carbon dioxide (CO_2) and nitrogen (N) as ammonia (NH_3) can confound moisture losses. Thus, the apparent moisture content (MC) that also includes C and N losses will be higher than the true MC. Further, use of apparent MC may also result in overestimation of C and N concentrations (dw basis). Broiler litter (BL) MC was determined using FD 50 (flask-dried at 50℃ for 24 h), FD 70 (flask-dried at 70℃ for 16h), CT70 (oven-dried at 70℃ to constant mass), and CT105 (oven-dried at 105℃ to constant mass). Carbon and NH_3-N losses during drying were measured for the FD50 and FD70 treatments. Carbon and N losses were significantly greater with FD70 vs. FD50; however, FD50 did not completely dry the BL and was hence, not appropriate for MC determination. Flask-drying and oven-drying at 70℃ were equally effective in completely drying BL. The true MC (22.5%) and apparent MC (26.3%) were significantly different. Hence, CO_2-C and NH_3-N losses accounted for 14.4% of the total mass lost during drying. While total Kjeldahl N (TKN), total ammoniacal-N (TAN), and total C (TC) concentrations calculated using apparent MC were 5% higher than the concentrations calculated using true MC, they were not significantly different. The TKN concentrations in the field-moist samples adjusted to dw basis for true or apparent MC were significantly greater than TKN concentration in CT105 possibly due to urea sublimation and decomposition at 105℃. Where accurate C and N accounting are required, C and N losses should be measured during drying. Due to wide variability in litter properties, extrapolating these findings to other litters or different sample sizes is not desirable.

Keywords: poultry litter, moisture content, NH_3 loss, CO_2 loss, urea

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1 Introduction

Since poultry litter (PL) is an important source of plant nutrients, analyses for MC, N, and phosphorus must be accurate and repeatable. Poultry litter is dried prior to sample analysis to minimize nutrient transformations and losses during storage and facilitate sample preparation (Wood and Hall, 1991). When comparing different litters, it is necessary to determine the MC of the samples to compare their chemical properties on dry-weight (dw) basis. Four methods of drying PL include oven-, air-, freeze- and microwave-drying (Sistani et al., 2001; Wood and Hall, 1991). However, freeze-drying is more expensive than oven-drying and also resulted in higher N losses than air- or oven-drying (Wood and Hall, 1991). Wood and Hall (1991) reported incomplete moisture removal with microwave-drying. Here, we will focus on forced-air or oven drying of poultry litter.

Many temperature-duration combinations have been used for MC determination of PL or poultry manure (PM). Pote et al. (2009) dried BL at 105℃ (duration unspecified)

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while Sistani et al. (2001) dried PM (from birds raised at three different temperatures) to constant mass at 65 ℃ and 105 ℃. Wood and Hall (1991) dried BL (two samples) at 40 ℃ (3 d) and 60 ℃ (3 d) using convective oven. For total solids (TS) determination in liquid manures, Wolf et al. (1997) recommended temperature-duration combinations of 110 ℃ - 6 h, 70 ℃ - 16 h and 50 ℃ - 24 h. Peters et al. (2003) reported that differences in residual MC values in all but PM were insignificant for samples dried at 50 ℃, 70 ℃, and 110 ℃, with the duration of drying varying with the sample size. It is unclear if Peters et al. (2003) used the term PM to describe PL because they may differ in terms of moisture removal. Obviously, the type of material and oven (forced convection vs. natural convection) also affect moisture removal. However, drying samples high in organic matter (OM) can cause thermal breakdown leading to loss of volatile constituents (Mills and Jones, 1996). Based on a review of literature, Gardner (1965) suggested that drying samples high in OM content at ≤50°C could minimize OM losses.

Drying BL can result in losses of both organic and inorganic N and C forms. Depending on the temperature and duration, NH_3-N, and C losses, and hence, their final concentrations in the litter will vary (Wood and Hall, 1991). Therefore, mass reduction during drying is not just due to moisture loss but also due to losses of C and N; this will result in overestimation of MC. Consequently, C and N concentrations (dw basis) in the litter may also be overestimated. While these inaccuracies may not be important for deciding land application rates, when accurate C and N mass balances are required, these small differences could be consequential. Therefore, direct measurement of CO_2-C and NH_3-N losses during the drying process may improve accuracy of measurement of litter properties. We are unaware of any work on measuring C and N losses during litter-drying for MC determination.

Hence, the overall objective of this research was to measure the impacts of drying temperature-time combinations on CO_2-C, and NH_3-N losses. Specific objectives were to: (a) determine NH_3-N and CO_2-C losses in broiler litter dried at 50 ℃ for 24 h and 70 ℃ for 16 h in flasks; (b) compare MC using the above treatments that provided complete moisture removal with the control treatments (oven-drying at 70 ℃ or 105 ℃ to constant mass); and (c) determine the error in calculating N and C concentrations in BL due to losses of N and C during drying. The treatments in specific objective (a) were based on Wolf et al. (1997), assuming that a drying treatment for liquid samples would also apply to solid BL samples because of their lower MCs. Moisture, N, and C losses were also measured by drying BL at 25 ℃ for 48 h, similar to air-drying (for 10 d) used by Wood and Hall (1991) but those results are not presented here because of incomplete moisture removal.

2 Materials and methods

Litter was obtained from a broiler house in Duplin Co., North Carolina (NC). The samples were placed in black plastic bags and transported on ice to the lab. After sieving (2 mm) and thorough mixing, the BL was analyzed in triplicate for TKN (APHA, 1998a), TAN (APHA, 1998b), and TC (EPA Method, 1974). Thereafter, the litter was frozen (-18 ℃) to prevent microbial activity until the start of experiments. Because of the wide variability in litter properties (NCSU, 2014), we focused on determining C and N losses from litter obtained from a single broiler house.

The four treatments were: (1) FD70: flask-drying at 70 ℃ for 16 h; (2) FD50: flask-drying at 50 ℃ for 24 h; (3) CT70 (Control 1): oven-drying at 70 ℃ to constant mass; and (4) CT105 (Control 2): oven-drying at 105 ℃ to constant mass. Each treatment was replicated three times and the BL was allowed to equilibrate to room temperature for 30 min before the experiment. In Treatments 1 and 2, in addition to moisture, CO_2 and NH_3 losses were also determined during drying and thus required a different experimental set-up (see below) vs. Treatments 3 and 4.

In FD70, and FD50, 50 g of the BL sample was placed in a 500 mL glass flask in a 0.5 cm layer; this flask was placed in a water bath to control temperature (70 ℃ or 50 ℃). Two scrubbers (polycarbonate flasks) each containing 250 mL of 2 molar (M) sodium hydroxide (NaOH) solution were placed downstream of the flask containing the litter to absorb CO_2 released by the litter during drying (Carter and Gregorich, 2006). Two scrubbers (efficiency >97%) (Shah et al., 2014), each containing 250 mL of 2% w/v boric acid (H_3BO_3)solution were placed after the CO_2 scrubbers to trap NH_3 (and amines) released by the litter during drying. Any CO_2 or NH_3 not trapped in the primary scrubber was assumed to be trapped in the secondary scrubber. Airflow through the system was provided by a vacuum pump and the airflow rate (118 mL/min) was controlled by a flow meter equipped with a needle valve (Model: GF-8321-1401, Gilmont Instruments, Barrington, IL). The pump was stopped for 30 s out of five minutes to prevent it from overheating.

Lab air was also sampled for CO_2 and NH_3 using a single scrubber for each gas. Blank samples were also used to adjust the NH_3 and CO_2 concentrations for contamination during solution preparation. Concentration of CO_2-C in the scrubber was obtained through titration (Carter and Gregorich, 2006) while scrubber NH_3-N concentration was determined using colorimetry on Auto Analyzer III. Based on the CO_2-C concentrations (mg/L) in both sequential scrubbers and their volumes (L), the mass of C trapped was determined. After adjusting for the ambient and blank CO_2-C (or NH_3-N) concentrations, the net CO_2-C (NH_3-N) mass loss from the litter was calculated. Since the NaOH scrubbers had an initial pH of 14.3, it was assumed that these scrubbers would not trap NH_3 and were thus, not analyzed for NH_3.

At the end of the drying period, the final litter masses were determined for the FD70 and FD50 treatments. For these two treatments, the net moisture loss was obtained by subtracting the masses of C and N trapped in the scrubbers from the mass lost during drying (initial minus final sample mass). True MC (dw) was calculated for FD70 by dividing the net moisture loss by the final BL mass. Apparent MC (dw) was calculated for FD70, referred to as FD70*, where the entire mass lost during drying was assumed to be moisture. Moisture content for the FD50 treatment was not calculated as it did not provide complete moisture removal (discussed later).

For CT70 and CT105, 50 g moist BL samples were dried in triplicate in a convection oven at 70 or 105 ℃, respectively, to constant mass. Since drying at 105 ℃ could cause more C and N losses than at 70 ℃, we also determined TKN, TAN, and TC concentrations in the dried samples using methods described earlier. We did not determine TKN, TAN, and TC concentrations in CT70 because it was only included to compare oven-drying (CT70) with flask-drying (FD70).

Treatment means for C, N, and moisture losses from FD70 and FD50 were compared using t-test in SAS (SAS, 2010). Moisture contents (dw) determined using FD70, CT70, and CT105 were compared using ANOVA; if at least one treatment was different, Tukey's Honestly Significant Difference (HSD) test was used to compare means (SAS, 2010). Since moisture loss from FD50 was much lower than FD70, FD50 was not compared with the CT70 and CT105 treatments. An alpha value of 0.05 was used throughout this study.

3 Results and discussions

Our field-moist BL samples had 30% and 40% lower TKN and TAN concentrations, respectively, than the average NC BL sample (Table 1) (NCSU, 2014). However, our TC concentration was 11% higher than the average NC BL sample (Table 1) (NCSU, 2014).

Table 1 Comparison of BL properties[1,2] for different treatments with published values

Treatment	Measured or calculated	TKN (mg/kg)	TAN (mg/kg)	Total C (%)
Published[3]	Measured	35,500 ±10,000[4]	6,035 ±416[5]	23.4 ±7.2[6]

Field-moist	Measured	24,797±655[7]	3,579±86[7]	26.1±2.1
CT105[8]	Measured	26,202±1,577b[9]	4,601±53	32.2±1.0
FD70*[10]	Calculated	33,646±888a[7]	4,888±117[7]	35.4±2.8
FD70[11]	Calculated	31,996±845a[7]	4,648±111[7]	33.7±2.6
p value[12]		<0.01[13]	0.05	0.29

Note: [1]All treatments except ‘Published’ and ‘Field-moist’ samples are on dw basis; MC values (not ‘Published’) are presented later. [2]Mean±SD for n = 3, unless indicated otherwise. [3]Based on samples submitted by broiler producers to the NC Dept. of Agriculture & Consumer Services (NCSU, 2014). [4]n = 1004 [5]n = 34. [6]n = 8. [7]n = 2. [8]Chemical properties of BL oven-dried at 105°C to constant mass. [9]Values followed by the same letter are not significantly different at α = 0.05 using Tukey’s honestly significant difference (HSD). [10]Chemical properties of field-moist samples adjusted to dw basis using average (n = 3) apparent MC (26.3%) determined using flask-drying at 70°C for 16 h . [11]Chemical properties of field-moist samples adjusted to dw basis using average (n = 3) true MC (22.5%) determined using flask-drying at 70°C for 16 h . [12]‘Published’ and ‘Field-moist’ samples excluded from statistical analyses. [13]Tukey’s HSD not calculated because of unbalanced data set.

Moisture loss was significantly higher in FD70 vs. FD50 (p < 0.01) (Figure 1). As discussed later, drying was complete with FD70 but not with FD50. As discussed later, flask-drying was as effective as forced-air drying in an oven at 70°C for 16 h. So, FD70 was effective in determining MC, even with a very large sample size of 50 g. Wolf et al. (1997) recommended drying liquid manure at 50°C for 24 h or 70°C for 16 to determine TS so it is surprising that BL MC determination was possible with FD70 but not with FD50. With samples sizes of ≤5 g, FD50 might have sufficed (Peters et al., 2003).

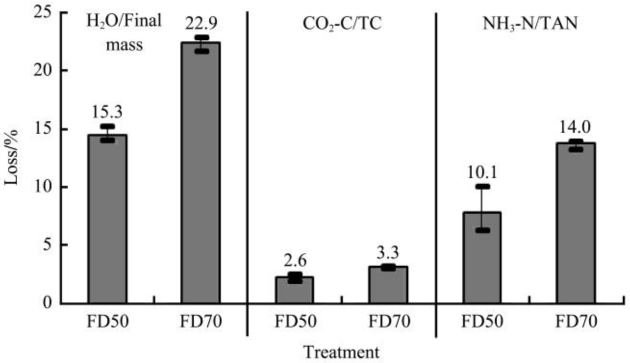

Figure 1 Comparison of moisture loss as percent of final litter mass, CO_2-C loss as percent of initial litter TC concentration, and NH_3-N loss as percent of initial litter TAN concentration between FD50 (flask-dried at 50 ℃ for 24 h) and FD70 (flask-dried at 70 ℃ for 16 h). Each value is a mean of three replications and the dashes show minimum and maximum values. The two treatments were significantly different in moisture (p<0.01), C (p<0.01), and NH_3-N (p = 0.01) losses at α = 0.05

Carbon loss (as percent of initial TC) was significantly higher in FD70 vs. FD50 (p < 0.01) (Figure 1). Carbon loss could be due to losses of volatile organic compounds (VOCs), microbial respiration (Atkinson et al., 1996), and release of dissolved CO_2 as the liquid fraction evaporated. Since only some of the released VOCs would have been retained in the NaOH scrubber, actual C losses (Figure 1) may have been underestimated. Based on the solubility of CO_2 in water (1.8 g/L at 25 ℃) (Carroll et al., 1991) and true MC of the samples, loss of dissolved CO_2-C was ≤1.5% of the total C loss at FD70 and probably lower from FD50. Despite possibly decreased microbial activity (due to high temperature and lack of moisture), greater C losses from FD70 could be due to greater dissolved CO_2 and VOC losses from FD70 vs. FD50.

Because dissociation of ammonium (NH_4) to NH_3 and H^+ (Jayaweera and Mikkelsen, 1990) and NH_3 desorption from the liquid phase (Dasgupta and Dong, 1986) increase exponentially with temperature, there was significantly (p = 0.01) greater NH_3-N loss (as percent of initial TAN) from FD70 vs. FD50. We assumed that no NH_3 would be trapped in the CO_2 scrubbers. Using Henry’s Law (assuming that the outlet gas concentration was the ‘equilibrium headspace concentration’) and with near-continuous airflow through the scrubbers, the highly-basic 2M NaOH solution would retain <<1% of NH_3 trapped in the acid scrubbers in the two treatments. The H_3BO_3 scrubbers may have trapped NH_3 formed due to thermal decomposition of urea (in TKN) (Paorici et al., 1995), particularly in the FD70 treatment. Because the FD50 treatment was ineffective in drying the BL samples, it will not be discussed further.

Apparent MC values determined using FD70* (flask-drying at 70 ℃ for 16 h), CT70 (oven-drying at 70 ℃ to constant mass), CT105 (oven-drying at 105 ℃ to constant mass) were not significantly different from one-another (Figure 2) but were significantly greater (p < 0.01) than the true MC (after adjusting for C and N losses) determined using FD70 (Figure 2).

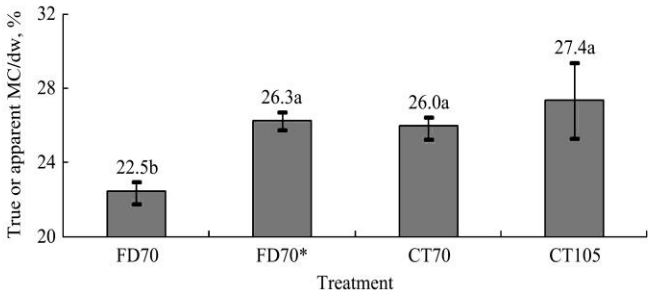

Figure 2 Comparison of MC (dw) among the treatments [FD70: flask-dried at 70 ℃ for 16 h, with C and N losses during drying considered (true MC); FD70*: flask-dried at 70 ℃ for 16 h but C and N losses during drying not considered (apparent MC); CT70 or CT105: oven-dried at 70 ℃ or 105 ℃ to constant mass, with C and N losses not measured during drying (apparent MC)]. Each value is a mean of three replications and the dashes show minimum and maximum values. Treatment means followed by the same letter are not significantly different at $\alpha = 0.05$ (Tukey's HSD = 3.0%)

Very close apparent MC values from the FD70* and CT70 treatments (Figure 2) showed that flask-and oven-drying were equally effective in drying BL. Based on the difference in MC between FD70 and FD70*, 14.4% of the reduction in mass during drying was due to C and N losses. Hence, true MC content was 85.6% of apparent MC. Oven-drying at 70°C for 16 h (Wolf et al., 1997) was appropriate for determining BL MC and there was no need to dry the samples at 105 ℃. While Peters et al. (2003) recommended drying PM at 110 ℃ for complete moisture removal, our 50-g BL samples were completely dried in 16 h at 70 ℃.

While CT105 concentration values were determined through analyses, the FD70 or FD70* concentration values (Table 1) were obtained by adjusting the field-moist chemical concentrations to dw basis, based on either true (22.5%) or apparent (26.3%) MC values, respectively. Despite a significant difference between true and apparent MC values (Figure 2), calculated chemical concentrations for any analyte for FD70* and FD70 were not significantly different from one another (Table 1). However, FD70 chemical concentrations were 5% lower than FD70* chemical concentrations (Table 1).

Both FD70 and FD70* had significantly ($p<0.01$) higher TKN concentrations than CT105 (Table 1), but TAN or TC concentrations were not significantly different among the three treatments (Table 1). Since the difference in TAN concentrations between FD70* and CT105 (both based on apparent MC) was very small (discussed later), the large difference in TKN concentration (22%) between those two treatments could have been due to thermal decomposition (and sublimation) of organic N compounds. In addition to sublimation, urea decomposes in the 80 ℃ to 100 ℃ range to form solid biuret and NH_3 (Paorici et al., 1995). Ghaedian and Wehling (1996) reported gaseous loss of some uric acid at 120 ℃, the lowest temperature that they tested, much lower than its melting point of 300 ℃.

Because NH_3 formed a much bigger fraction of TAN than TKN, NH_3 volatilization should have resulted in much (perhaps, significantly) higher TAN loss from CT105 than FD70* or FD70. Due to high ionic concentration and NH_4 adsorption onto the solid matrix, the dissociation constant of NH_4 in BL slurry ($K_d' = 1.02\times10^{-10}$ at 25 ℃) (Equation (1)) was only 18% of its dissociation constant in aqueous solution (K_d) (Liang et al., 2014).

$$K_d' = \frac{[\mathrm{NH_3\text{-}N}]\{\mathrm{H^+}\}}{\{NH_4\text{-}N\}} \qquad (1)$$

In Equation (1), the species inside curly brackets denote ionic concentrations, which would be lower than the stoichiometric concentrations (square brackets) in BL slurry.

At 25 ℃, with pH of 7.3 (measured in moist BL), using Equation (1), only 0.2% of the TAN would have been in the NH_3 form. Since K_d' increases exponentially with temperature (Jayaweera and Mikkelsen, 1990), at 100 ℃, 16% of the TAN would have been present as NH_3. However, NH_3-N loss should have been >16% of TAN from CT105 because with increased temperature (and reduced solvent mass), the dissolved NH_4 should have rapidly converted to NH_3 and volatilized, causing more adsorbed NH_4 to solubilize, convert to NH_3, and volatilize. But the TAN concentrations between FD70* and CT105 (Table 1) differed by only 6% (not significant); as expected, difference in TAN concentration between FD70 and CT105 was only 1% (Table 1). In fact, the ratio of TAN to TKN actually increased from 14.4% for the field-moist BL to 17.6% for CT105 (Table 1), which was unexpected. The smaller loss in TAN concentration during drying was probably because K_d' measured in BL slurry (Liang et al., 2014) and used in this study, was too high for use in unsaturated BL. However, currently, there is no instrument available to measure dissolved NH_3 concentration in the liquid film of unsaturated BL samples.

Lack of significant difference in TC concentrations ($p = 0.29$) among CT105, FD70, and FD70* may have been due to greater within-treatment variability in the FD70 and FD70* (Table 1) which might have masked treatment effects. It is also possible that organic compounds lost during drying (CT105) had more N than C. For example, if a mole of urea is sublimated or decomposed, 28 g of NH_3-N will be lost vs. only 12 g of C.

Our findings are consistent with both Wood and Hall (1991) and Sistani et al. (2001), who reported lower TKN, TAN, and TC (only Wood and Hall, 1991) concentrations in the dried manure or BL samples than in the moist samples adjusted to dw basis. Difference in total N loss (as percent of FD70*) between FD70* and CT105 were comparable to Sistani et al. (2001) but the much-lower difference in TAN loss in this study could be due to much higher manure TAN concentrations in Sistani et al. (2001). On a percent basis, Wood and Hall (1991) reported lower total N loss but higher TAN loss when BL samples were oven-dried at 60 ℃ for 3 d (OD60) vs. this study. Higher TAN losses in Wood and Hall (1991) were due to much higher TAN concentrations and perhaps, longer duration of drying vs. this study. Despite lower TC concentrations, Wood and Hall (1991) reported a significant reduction in TC during drying but the TC reduction was not significant in this study which may be due to the longer duration of OD60 vs. this study which allowed greater C loss due to (thermophilic) microbial respiration (Atkinson et al., 1996).

Field moist NC BL properties vary widely in their chemical propertiese.g., TKN (7,500 to 66,500 mg/kg, n = 1,004), TAN (7 to 36% of TKN, n = 34), TC (116,060 to 317,791 mg/kg, n = 8), and MC (3 to 39%, n = 1,023, wet basis) (NCSU, 2014). While we have demonstrated that C and N losses accounted for 14.4% of the mass lost while drying BL, readers are cautioned against applying our findings directly to their litters because litter properties will also affect the difference between apparent and true MC values. In litter samples with higher C or N concentrations (vs. this study), due to greater gaseous C or N losses, true MC may be a smaller fraction of the apparent MC. The reverse may be true for samples with lower C or N concentrations than this study. When more accurate C and N accounting is required, their gaseous losses should be measured during drying.

4 Conclusions

The following conclusions can be drawn from the drying experiments for broiler litter:

1) Losses of CO_2-C and NH_3-N increased significantly with drying temperature.

2) Moisture content of up to 50 g samples of BL can be determined satisfactorily by drying at 70 ℃ for 16 h. Drying BL at 50 ℃ for 24 h did not result in complete moisture removal.

3) Losses of NH_3-N and CO_2-C during drying at 70 ℃ for 16 h accounted for 14.4% of the mass lost. Hence, true MC was 85.6% of apparent MC. In litter samples with higher C or N concentrations, true MC as percent of apparent MC may be lower than in this study due to greater C or N loss.

4) While average TKN, TAN, and TC concentrations calculated using apparent MC where 5% higher than the concentrations calculated using true MC, they were not significantly different. However, the TKN concentrations in the field-moist samples adjusted to dw

basis for true or apparent MC were significantly greater than TKN concentration in CT105.

5) Drying at 105℃ resulted in substantial TKN loss, possibly, due to urea sublimation and decomposition.

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