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ARTICLE

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Evaluation of biosolids-enriched ammonium sulfate vs. ammonium sulfate in soils and for plant growth

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Abstract

Nitrogen (N) and sulfur (S) are essential nutrients for optimum plant growth. A commercial-grade biosolid-enriched ammonium sulfate (Bio/AMS) was evaluated and compared with a conventional, commercial-grade ammonium sulfate (AMS) to investigate the release patterns of N and S in soils. The laboratory-based studies included (a) characterization of fertilizer sources, (b) soil leaching columns with 0.01% citric acid in an acid soil, (c) soil incubation followed by 1 M KCl extraction in a neutral soil, and (d) NH_3 volatilization measurements in an alkaline soil. With one exception, the results showed no significant difference in the amount or the rate of N or S released from each between the two products in all the studies. In that one exception, the Bio/AMS leached significantly less N after 56 d, but not before 14 d, than the conventional AMS application. In greenhouse-based evaluations, the Bio/AMS and AMS performed comparably at providing S nutrient to the biomass yield and S uptake by canola (*Brassica napus* L.) at maturity in neutral and alkaline soils. The studies also showed no significant N uptake differences between Bio/AMS and AMS by wheat (*Triticum aestivum* L.) biomass at maturity in acid and neutral soils. The results from these laboratory and greenhouse studies demonstrated that the Bio/AMS product is a simple physical mixture of biosolids with AMS.

1 | INTRODUCTION

Nitrogen (N) and sulfur (S) are essential nutrients for optimum plant growth (Black, 1968). While N fertilizers have long been used to provide N for crop production, S fertilizers have become increasingly prevalent in recent years (Jibiao et al., 2016). Due to the reduction in SO_2 emissions from industry in response to environmental regulations on the prevention of air pollution, S deficiency in agricultural soils has become a limiting factor for crop yield in many countries (David et al., 2016). Many popular NPK fertilizers contain little or no S. Traditionally, the major S sources have been single superphosphate (SSP), which contains gypsum ($CaSO_4$), ammonium sulfate (AMS), and elemental sulfur (ES) (Chien et al., 2009). Natural gypsum and phosphogypsum (a by-product of H_3PO_4 production) are used as soil amendments and supply S nutrient as well. It should be noted, however, that ES does not provide plant-available S until it is oxidized to the SO_4–S form in soils (Chien et al., 2011, 2016).

Several new NP fertilizers containing ES plus AMS have been introduced to the market. The AMS provides available S immediately after planting, whereas ES oxidation provides available S at later stages of crop growth. A review of the agronomic effectiveness of AMS vs. S fertilizer sources containing AMS with or without ES conducted by Chien et al. (2016) concluded that the granular form of ES may not be sufficiently oxidized to benefit crop growth within the season of

Abbreviations: AMS, ammonium sulfate; Bio/AMS, biosolids-enriched ammonium sulfate; ES, elemental sulfur.

its application. Even after granule disintegration, the localized ES particles around the applied granule sites limit the surface area coming into contact with soil and oxidizing microbia, thus limiting ES oxidation as compared with the application of powdered ES particles to the soil. Consequently, for the first crop or during the season following fertilizer application, available S is derived mostly from the AMS component, with the associated micronized ES particles contributing only an insubstantial amount of the available S. Furthermore, granular NP products containing ES and AMS such as MAP-(5% ES + 5% AMS) often do not perform well when compared with traditional SO_4–S based products like SSP, AMS, and gypsum (Chien et al., 2016; Casteel et al., 2019).

Recently, a granular biosolid-enriched ammonium sulfate (Bio/AMS) stating to be a slow N- and S-release fertilizer has entered the market. Performance data is limited for this new product, but at least one recent report has demonstrated that there is no difference in yield of flooded rice (*Oryza sativa* L.) when the crop was fertilized with this Bio/AMS as compared with conventional AMS (Harrell, 2018).

To address the lack of information regarding this new fertilizer, we undertook the work of this paper to compare a commercial-grade product of Bio/AMS with AMS in the following studies: (a) characterize the chemical composition of the Bio/AMS material, (b) examine the movements of N and S through soil leaching columns, (c) determine N release over time using soil incubation studies, (d) examine ammonia (NH_3) volatilization as affected by the sources, and (e) evaluate the availability of S and N for plant growth in a greenhouse study conducted with canola (*Brassica napus* L.) and wheat (*Triticum aestivum* L.).

Core Ideas

- Nitrogen and S are essential nutrients for plant growth.
- Ammonium sulfate provides both N and S nutrients.
- Can enriched biosolids enhance N and S availability of ammonium sulfate?

2 | MATERIALS AND METHODS

2.1 | Characterization of fertilizer sources

This study included commercial-grade granular Bio/AMS fertilizer (trade name SymTRX, Anuvia Company) labeled with an analysis of N–P–K–S–Fe as 16–0.4–0–20–2 with 16% organic matter. This product was compared with a conventional, commercial-grade granular AMS fertilizer labeled with an analysis of N–P–K–S as 21–0–0–24. The source and composition of the biosolids used in the Bio/AMS product, however, were not provided by the producer.

Representative granular samples of the Bio/AMS (2.8–3.0 mm) and AMS (2.0–2.8 mm) products were finely ground to 2-mm size. Three ground samples of each source were analyzed by water extraction (1 g per 100 ml) for soluble SO_4–S on an inductively couple plasma-optical emission spectroscopy (ICP-OES), and for soluble total N and NH_4–N on a Skalar Segmented Flow Analyzer. The total N content was analyzed using the Leco CN628 and the total S was analyzed using the Leco SC144DR.

2.2 | Soil leaching column study

The slow-release characteristics of the N and S were measured following the AOAC-adopted soil leaching column technique (Medina et al., 2014). Incubation lysimeters were constructed from cylindrical PVC pipe measuring 30 cm in length by 7.5 cm in diameter. A piece of landscaping fabric was placed in the bottom of the column and held in place with the bottom pipe cap to prevent solids from leaching out of the column. Approximately 90 g of collected top soil (Ultisol) from eastern Lauderdale County in Alabama and 1,710 g of quartz sand were thoroughly mixed and placed into a column. The soil had been air-dried and screened to 2 mm size before mixing. Two pertinent soil properties were pH of 5.3 and 65 g kg^{-1} of organic matter. The amount of total N applied from Bio/AMS and AMS incorporated into each column was 450 mg N while the amount of associated total S applied was 490 mg S from Bio/AMS and 503 mg S from AMS, respectively. Three replicates of soil columns were set up for each treatment.

Columns were leached by pouring 500 ml of 0.01% citric acid solution into each column on Days 7, 14, 28, and 56. The use of the 0.01% citric acid was intended to simulate the soil's natural biological activity (Medina et al., 2014). A filter flask connected to the bottom of each column with a ball valve and the column was allowed to flow by gravity for 5 min into the flask by opening the ball valve. After 5 min, a vacuum hose was attached to each flask and vacuum was pulled for 2 min. Then vacuum was removed, ball valve was closed, and top cap was replaced on each column. The solution volume was recorded and then transferred to a 500-ml bottle for storage. The leachate samples were analyzed for total S by ICP and for total N, NH_4–N, and NO_3–N on a Skalar Segmented Flow Analyzer.

2.3 | Soil incubation study

The top layer (0–15 cm) of a Norfolk loamy sand (fine-loamy, kaolinitic, thermic Typic Kandiudult) collected from Alabama was used. Soil was air-dried and ground to pass

through a 2-mm screen. The soil had a pH of 6.2 and organic matter of 50 g kg^{-1}.

To initiate the incubation study, 9 ml of water was first added to an empty 236-ml plastic specimen container (Fisher Scientific) followed by the addition of 100 g of soil which had been thoroughly mixed with 250 mg N kg^{-1} from the granular Bio/AMS or the AMS sources. Another 9 ml of water was then spread over the soil surface to attain 82% of soil field capacity. A control (no N) was also included. The weight of each container (without lid) was recorded. Three replicates were set up for each treatment.

Each container was covered by a lid with 20 small punched holes for aeration during the incubation at 30 ˚C for 0, 1, 2, 3, 4, 6, 7, 8, and 10 wk. The soil moisture of each container was adjusted by weighing without the lid, and adding water as needed to maintain 82% of soil moisture field capacity twice weekly. At the end of each incubation period, 200 ml of 1 M KCl was added to each container, and the punched lid was replaced with a lid with no holes. The containers were shaken for 1 h followed by filtration. Extractable NH_4–N and NO_3–N were determined by a Cd reduction method (Wood et al., 1967) followed by colorimetric analysis.

2.4 | Ammonia volatilization study

The soil used was collected from the top layer of a Brownfield loamy sand (loamy, mixed, superactive, thermic Arenic Aridic Paleustalf) from Texas with the following soil properties: pH = 8.2, CEC = 6.92 cmol kg^{-1}, P-extractable SO_4–S = 2.81 mg S kg^{-1} and organic C = 5.1 g kg^{-1}. Soil pots (8 kg) were kept at 75–80% field capacity for 1 wk before fertilizer application. Fertilizer granules from Bio/AMS, AMS, or urea were evenly distributed on the soil surface at a rate of 200 kg N ha^{-1}. A control without N was included in the treatments, which were replicated three times.

The pots were tightly sealed from the ambient air, but with air inflow from a compressed air system and outflow to the H_3PO_4 acid traps. The air from the compressed air system was passed through an acid scrubber to remove any NH_3 in the air and humidified through a water bottle before entering the pots. The airflow rate was adjusted to 5 L min^{-1} for each pot to allow for complete mixing with the air and maximum removal of any NH_3 formed. All NH_3 (outflow from the pots) was captured in the acid traps (Stumpe et al., 1984). The time intervals for NH_3–N collection were 1, 2, 3, 5, 7, 10, 13, and 16 d.

Solutions from the acid traps were analyzed for NH_4–N with colorimetric method as described by Mulvaney (1996). The automated procedure for NH_4–N determination was based on the modified Berthelot reaction (Krom, 1980; Searle, 1984). The spectrophotometric analysis of the ammonia complex was conducted at 680 nm.

TABLE 1 Pertinent soil properties of the soils used in greenhouse study

Crop	Soil	pH	CEC	SO_4–S	Organic C
			cmol kg^{-1}	mg S kg^{-1}	g kg^{-1}
Canola	Brownfield	8.2	6.9	2.8	0.5
	Lakeland	5.8	0.8	5.7	0.6
Wheat	Ashland	4.9	11.7	–	1.4
	Manhattan	6.5	15.6	–	2.5

Note. CEC, cation exchange capacity.

2.5 | Greenhouse evaluation

The soils used in the canola experiment were the same Brownfield soil from Texas used in the NH_3 volatilization study and a Lakeland sandy soil (thermic, coated Typic Quartzipsamment) collected from Georgia. Pertinent characteristics of these two soils were shown in Table 1. Non-draining pots were filled with 8 kg of each soil, which was thoroughly mixed with either the Bio/AMS or the AMS fertilizer at rates of 0, 5, 10, 20, and 40 mg S kg^{-1}. All other nutrients including NPK and micronutrients were adjusted and added to all the treatments at the same adequate levels so that S was the only limiting factor to canola growth. About 10–12 seeds of canola were planted and thinned to two plants after germination. The soil moisture was maintained at approximately 80% field capacity by daily watering during plant growth.

The soils that were used for the wheat experiment were Smolan silty clay loam (fine, smectitic, mesic Pachic Argiustoll) from Manhattan, KS, and Stonehouse (sandy, mixed, mesic Typic Udifluvent) from Ashland Bottoms, KS. Details on these soils are included in Table 1. Non-draining pots were filled with 8 kg of each soil, which was thoroughly mixed with rates of 0, 25, 50, 100, and 200 mg N kg^{-1} of Bio/AMS or AMS. All other essential nutrients were added and adjusted at the same adequate levels so that N was the only limiting factor to wheat growth. Wheat (variety Zenda, DVP number: 201700282) seeds were vernalized at 4 ˚C. These seeds were then planted onto germination trays in a greenhouse for 1 wk and the resulting seedlings were placed in a vernalization chamber for a period of 8 wk at 4 ˚C and 100% relative humidity for a minimum of 6 wk. After vernalization, the plants were transplanted to five plants per pot. Eight days later, the plants were thinned down to three plants per pot. The soil moisture was kept at approximately 80% of field capacity by daily watering during plant growth.

Three replicates were set up for each treatment in the canola and wheat experiments. Wheat and canola plants were harvested at maturity. The grain and straw of wheat and the seed, pod shell and straw of canola were separately collected, dried at 60 ˚C, weighed, and ground to 0.5 mm. The wheat plant

TABLE 2 Chemical composition of analyzed biosolids-enriched ammonium sulfate (Bio/AMS) and ammonium sulfate (AMS) products

Product	Total N	NH_4–N[a]	Total S	SO_4–S[a]	NH_4–N/SO_4–S
		%			
Bio/AMS	17.2	16.4	20.1	19.3	0.85
AMS	20.4	19.7	22.8	22.5	0.87

[a]Water soluble.

samples were wet digested and N concentrations were determined by colorimetric analysis. The S concentrations of the canola plant were determined by dry combustion (Leco Analyzer) (Jones & Isaac, 1972).

2.6 | Statistical analysis

Statistical procedures were carried out with the software (Analysis ToolPack) in Excel for Windows 10. Analysis of variance on two-factor with replication was performed to detect significant effects ($p = .05$) for the source, time and source x time treatments in the laboratory experiments. In cases where there were significant differences in source treatments, values of Fisher's least significant difference (LSD .05) were used to compare the means between sources at a given time. In the greenhouse experiments, ANOVA was performed for the source, rate, and source x rate interaction treatments. In cases where there were no significant differences ($p = .05$) among source treatments, a suitable common regression function was fitted into the data to describe the relationship between the plant biomass yield or nutrient uptake and the rate of N or S applied from each source.

3 | RESULTS AND DISCUSSION

3.1 | Characterization of fertilizer sources

The labeled N and S contents of the commercial-grades of Bio/AMS and AMS fertilizers were 16N–20S and 21N–24S, respectively. The analyzed grades were 17N–19S and 20N–23S for the Bio/AMS and AMS products, respectively (Table 2). Water-soluble NH_4–N and SO_4–S (16.4% N and 19.3% S) in Bio/AMS were lower than that of AMS (19.7% N and 22.6% S) due to the dilution effect of 16% biosolids in the Bio/AMS product. The amount of water-insoluble organic N in the biosolids was determined by substracting the amount of water-soluble NH_4–N from total N as 0.8% (i.e., 17.2 – 16.4) (Table 2), which number approximates the 1% that was labelled on the Bio-AMS product. Therefore, the organic N component played no discernable role in the Bio/AMS product; and all impact of N should be attributed to the water-soluble NH_4–N form.

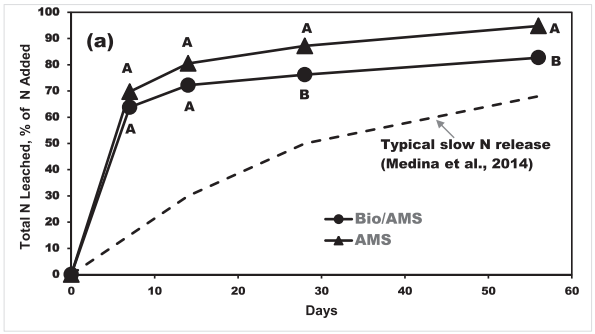


FIGURE 1 Leaching of (a) total N and (b) NH_4–N as % of toal N added from biosolids-enriched ammonium sulfate (Bio/AMS) and ammonium sulfate (AMS) in soil columns with 0.01% citric acid. Values of data points of Bio/AMS and AMS on the same day with the same letter were not significantly different from each other ($p = .05$)

The ratio of water-soluble NH_4–N/SO_4–S, which was 0.85 for Bio/AMS and 0.87 for AMS (Table 2), strongly suggests that the major water soluble N and S components of the Bio/AMS product were in the same form as in the AMS. In other words, the Bio/AMS product was simply a physical mixture of AMS and the biosolids. Furthermore, there is no indication that the NH_4–N complexed to any extent with the organic matter of biosolids in the Bio/AMS product. This is because the complexed NH_4–N in exchangeable NH_4–N form is not water soluble. It can be extracted only with ionized salt solutions such as commonly used KCl or $CaCl_2$.

3.2 | Soil leaching column study

There was significantly lower total N leached out from Bio/AMS than that from AMS after 14 d (Figure 1a). After 56 d, 83% of the total N from the Bio/AMS treatment had been leached through the soil column, as compared to 95% from the AMS treatment. Figure 1b shows that NH_4–N leached out from Bio/AMS was significantly lower (12%) than that from AMS after 14 d. One possible explanation for the lower amount of N leaching out from the Bio/AMS product than

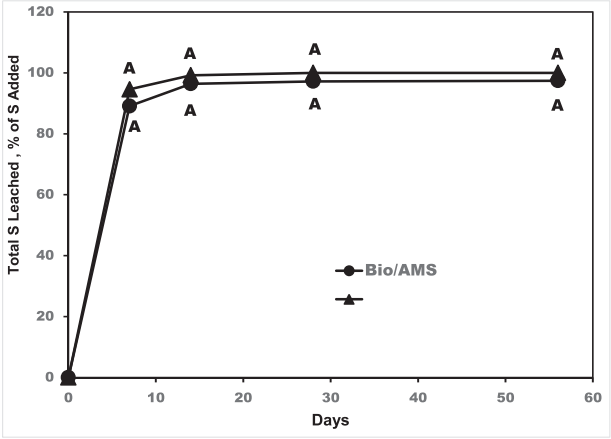


FIGURE 2 Leaching of total S from biosolids-enriched ammonium sulfate (Bio/AMS) and ammonium sulfate (AMS) in soil columns with 0.01% citric acid. Values of data points of BIO/AMS and AMS on the same day with the same letters were not significantly different from each other ($p = .05$)

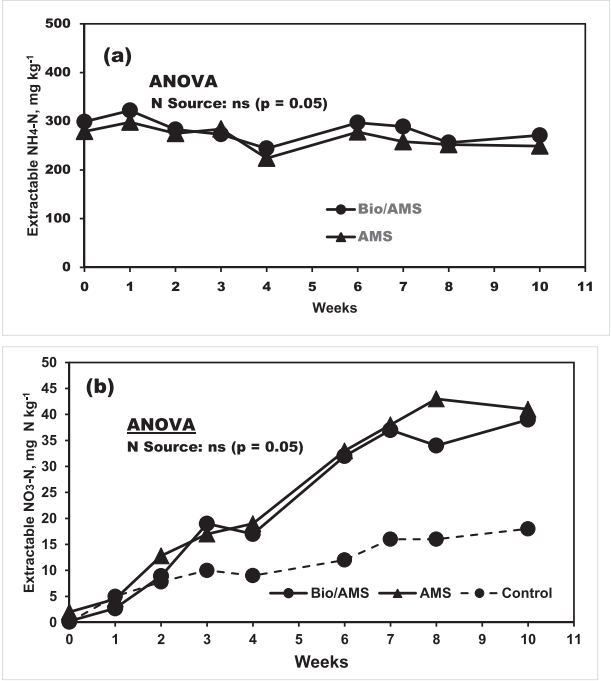


FIGURE 3 Amounts of KCl-extractable (a) NH_4–N and (b) NO_3–N from biosolids-enriched ammonium sulfate (Bio/AMS) and ammonium sulfate (AMS) during soil incubation with Norfolk soil (pH 6.2)

from the AMS product could be microbial N assimilation as a result of the 16% biosolids mixed in the former, thus increasing the C/N ratio around the Bio/AMS granules and enhancing soil N immobilization of NH_4–N (Black, 1968). During the leaching period, only small amounts of NO_3–N were found in the leachates from both Bio/AMS and AMS (data not shown). By 56 d, 4.7 and 5.1% of total N applied were leached out as NO_3–N for AMS and Bio/AMS, respectively. This indicates that only a low degree of nitrification from NH_4–N to NO_3–N with both products occurred during the leaching period.

Nitrogen release pattern of a typical slow-release N fertilizer as measured by the same AOAC-adopted soil leaching column technique that was developed and reported by Medina et al. (2014) is shown in Figure 1a. It shows that in the first 7 d, only 15% of total N was released from the typical slow-release N fertilizer as compared to 60% from Bio/AMS. At 28 d, only 50% of total N was released from the typical slow-release N fertilizer whereas 78% was released from Bio/AMS. Furthermore, a typical slow-relase N fertilizer continuously slowly releases N from 7 to 56 d without reaching the plateau, in contrast, the Bio/AMS appeared to attain the maximum release between 26 and 56 d (Figure 1a). Therefore, Bio/AMS, like AMS, does not fit the release pattern from a typical slow-release N fertilizer as described by Medina et al. (2014).

Figure 2 shows that the tested Bio/AMS and AMS followed the same S release patterns during the entire leaching period. Both products released 95% of total S in 7 d and almost 100% thereafter. Since the S form of both Bio/AMS and AMS was water-soluble SO_4–S (Table 1) and there were no differences in S leaching in the soil columns with 0.01% citric acid (Figure 2), it is clear that SO_4–S did not complex with the organic matter of biosolids in the bio/AMS product.

3.3 | Soil incubation study

There were no significant differences in KCl-extractable NH_4–N from Bio/AMS and AMS (Figure 3a) and the amounts of NH_4–N extracted from the non-treated soil (control) were insignificant (data not shown) during the incubation period. The stable trend of extractable NH_4–N for both N sources suggest that there was no significant nitrification of NH_4–N to NO_3–N, probably due to inadequate aeration during the soil incubation, even though 20 small holes had been punched on the lids for that purpose. This was supported by the relatively small increase in NO_3–N for both N sources during the soil incubation (Figure 3b). By subtracting the amount of NO_3–N of the control (18 mg N kg^{-1}) from the amount of the two products (41 mg N kg^{-1}) at the end of 10-wk incubation, there was only a small amount of NO_3–N (23 mg N kg^{-1}) derived from the total 250 mg N kg^{-1} applied with either Bio/AMS or AMS. This may explain as why the amounts of NH_4–N in the soils treated with Bio/AMS or AMS remained relatively constant during the incubation time (Figure 3a).

The results from this soil incubation study show that Bio/AMS and AMS were similar in N release and transformation in the soil. Like AMS, Bio/AMS did not behave as a slow N-release fertilizer in the soil.

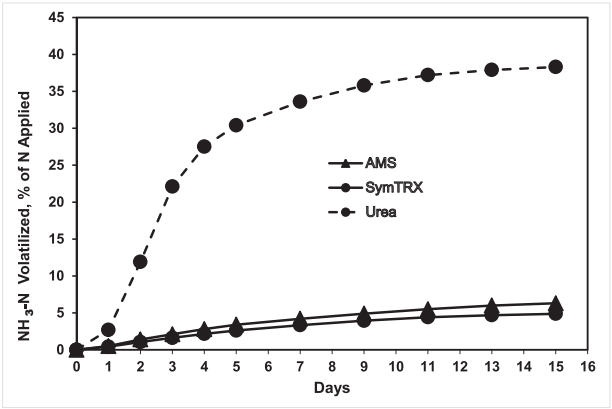


FIGURE 4 NH_3–N volatilized from urea, biosolids-enriched ammonium sulfate (Bio/AMS) and ammonium sulfate (AMS) on Brownfield soil (pH 8.2)

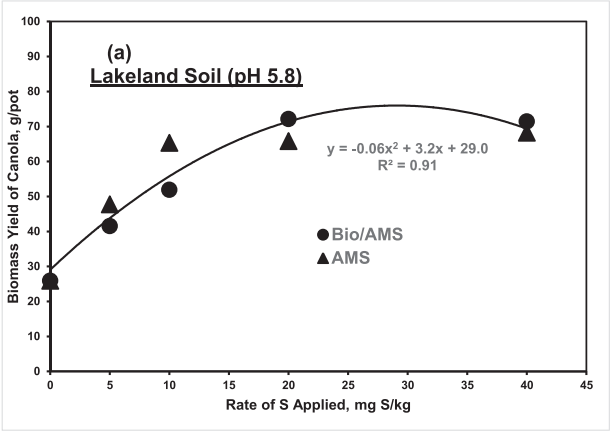
3.4 | Ammonia volatilization study

The amounts of NH_3–N volatilized by the end of the study were <5% of total N applied when both the Bio/AMS and AMS products were applied to the alkaline Brownfield soil (pH 8.5) whereas 35% of total N volatilized from the urea-N treatment (Figure 4). There were no significant differences in the amounts of NH_3–N volatilized from Bio/AMS vs. AMS from both products. The low amounts of NH_3–N volatilized from AMS and Bio/AMS were due to the acidic nature of AMS (Chien et al., 2011). It suggests that NH_4–N of AMS did not chemically complex with biosolids within the Bio/AMS matrix and both the AMS and Bio/AMS products behaved similarly in NH_3 volatilization on the alkaline soil.

3.5 | Greenhouse evaluation

In the canola experiment, canola seed yields were inconsistent among the samples tested against the various S sources, and therefore a clear conclusion cannot be drawn. It was observed that abortion rates during flowering among treatments were also inconsistent, probably due to the unusually cold 2020 winter in northern Alabama. However, total aboveground biomass yield data was more consistent within treatments and replicates.

Based on ANOVA, there was a significant canola biomass yield response to S in the sandy Lakeland soil. However, there were no significant differences in canola biomass yield between Bio/AMS and AMS (Figure 5a). Similarly, there was a significant canola biomass yield response to S but no significant difference between the two S sources in the alkaline Brownfield soil (Figure 5b). Canola biomass yield responses to S from the Bio/AMS and AMS products can be described by the same quadratic functions for the two soils (Figure 5).



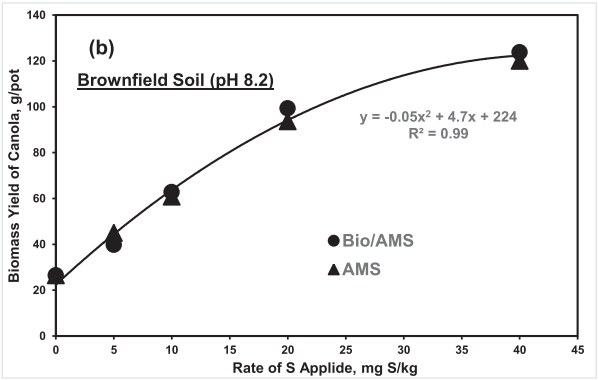


FIGURE 5 Biomass yield of canola obtained with biosolids-enriched ammonium sulfate (Bio/AMS) and ammonium sulfate (AMS) in (a) Lakeland soil and (b) Brownfield soil

Sulfur uptake by canola biomass also showed no significant differences between the two S sources for both soils (data not shown). These results clearly indicate that Bio/AMS and AMS were equally effective in providing S nutrient to the canola crop grown to maturity.

In the wheat experiment, the total biomass yield of wheat treated with N was very low (mostly <2 g per pot) for both soils, irrespective of N source and N rate (data not shown). This was likely due to an extraordinarily cold 2020 winter in Kansas, depressing wheat growth in general. According to Black (1993), plant nutrient uptake can be a reliable test when comparing nutrient sources under conditions where no significant or weak yield response is observed. Baed on ANOVA, a significant wheat response to N rate was observed in terms of N uptake by wheat biomass for both soils and N sources (Figure 6). However, there were no significant differences between the wheat responses of the two N sources as described by the same curvilinear response function for Ashland soil (Figure 6a) and the same linear response function for Manhattan soil (Figure 6b). These results indicate that Bio/AMS and AMS performed equally effective in providing available N to the wheat crop grown to maturity.

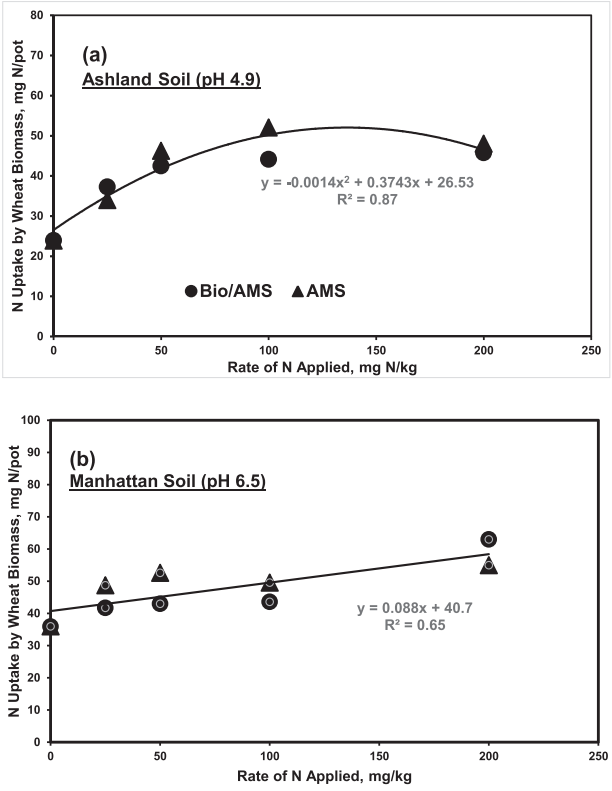


FIGURE 6 Nitrogen uptake by wheat biomass from biosolids-enriched ammonium sulfate (Bio/AMS) and ammonium sulfate (AMS) in (a) Ashland soil and (b) Manhattan soil

4 | CONCLUSION

A commercial-grade biosolids-enriched AMS product was evaluated against a conventional AMS to compare their release rates of total N and S in laboratory studies. The results from the soil-leaching columns, soil incubation and NH_3 volatilization experiments showed that there were no significant differences in the rates of N and S release between the two products except that Bio/AMS released less total N than AMS did in the leaching column study. The lower leached N rate from Bio/AMS was probably due to soil N immobilization as induced by the enriched biosolids in the Bio/AMS product. The results from the greenhouse experiments showed no significant differences in biomass yield of canola and S uptake at maturity in response to the two different products serving as S source in two different soils (neutral and alkaline). The results also showed no significant differences in N uptake by wheat biomass at maturity in response to the two different products as N source in two soils (acid and neutral).

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