

## Increasing Soil Organic Matter Content in Mine Soil through Pig Manure Addition

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### ABSTRACT

Mine soils in southeast Spain have scarce vegetation due to very poor properties such as extremely low soil organic matter (SOM) (< 0.6 g carbon kg<sup>-1</sup> soil), low pH, high salinity and metal contents. Also in southeast Spain, there is an economically-important pig husbandry faced with challenges to manage the large volumes of industry-generated animal wastes. This study will present the results of a leaching experiment to assess the retention and release of nitrogen and carbon from pig manure added to undisturbed column of mine soil. We excavated three columns (15-cm diameter and 30-cm length) from a representative mined site. The columns were amended with single (7 % by mass) and double doses of pig manure, and leached weekly with distilled water for 10 weeks to simulate annual rainfall events in the study area. Leachates were collected and analyzed for pH, electrical conductivity, redox potential, and contents of selected anions and metals. However, we will limit this presentation to carbon and nitrogen to quantify the potential contribution of pig manure addition to the build up soil organic matter in mine soils. Results showed that after addition of pig manure in the soil surface, soil pH increased from 2.2 to 4.0 after 11 weeks (single dose) and to 5.2 at week 21 (double doses). Significant increases were observed in total nitrogen contents in both single and double doses, 1.14 g kg<sup>-1</sup> (900 %) and 1.40 g kg<sup>-1</sup> (1100 %), respectively. Total carbon contents increased to 18.6 g kg<sup>-1</sup> (3200%) in single dose and to 16.4 g kg<sup>-1</sup> (2800%) in double doses. Nitrogen and carbon in soils had weekly rate of increases of 0.1 and 2.0 g kg<sup>-1</sup>, respectively. Moreover, C/N ratio increased from 5 to 12 at the end of the experiment. Leachates had significantly higher weekly release of NO<sub>3</sub><sup>-</sup> than total dissolved organic carbon (DOC) during the first 6 weeks of leaching. Weekly rate of releases (mg L<sup>-1</sup>) were 127 (NO<sub>3</sub><sup>-</sup>) and 5.2 (DOC) in single dose, and 35 (NO<sub>3</sub><sup>-</sup>) and 2.8 (DOC) for double doses. Leachates contained NO<sub>3</sub><sup>-</sup> less than the 50 mg L<sup>-1</sup> threshold established by FAO. These results suggest that addition of pig manure may significantly accelerate the build up of SOM in mine soils without endangering the release of NO<sub>3</sub><sup>-</sup> into sub-soil or groundwater in semiarid regions. Once there is sufficient SOM, mine soils will have an environment hospitable to various ecosystems including plant colonization and microbial community needed for its physical stability. Pig manure amendment of mine soils can be an ecologically-sound means of managing the large volume of wastes generated by the pig industry in southeast Spain

**Keywords:** Mine soils, TOC mine soil, nitrogen, leaching columns experiment

## INTRODUCTION

Mine tailings stored in mine tailing pond are residues from the extraction of lead/zinc (Pb/Zn) generated during the long history of mining activities in Murcia - southeast Spain. Surficial storage of these materials poses environmental risks from wind and water erosions, runoff and potential leaching of metals into the groundwater. An effective method to stabilize these materials is to establish a permanent plant cover. However, soils in mine tailings are difficult to revegetate due to nutrient deficiencies and metal toxicities (Bradshaw and Johnson, 1992). Due to the lack of organic matter and severe nutrient limitations of mine tailings, an amendment of pig manure could ameliorate mine soil by providing available nutrients (e.g., nitrogen), improve physical properties and possibly lower the availability of toxic metals through complexation with the organic matter (Ye et al, 2002).

Organic matter improves retention of nutrients by increasing cation exchange capacity, enhances the availability of nutrients (e.g.,  $\text{NH}_4^+$ ,  $\text{NO}_3^-$ ,  $\text{PO}_4^{3-}$ ,  $\text{SO}_4^{2-}$ ) and trace elements by mineralization, improves soil buffering capacity, chelates metallic ions increasing the availability of some nutrients, and decreases the toxicity of other ions. (McBride, 1994). Organic amendments are commonly added to disturbed sites to provide a source of N and encourage mineralization for crop growth. Nitrogen is frequently the most limiting nutrient for crop production on mine soils (Roberts et al., 1988), so the nitrogen mineralization potential of a soil is an indicator of fertility and site productivity. Nitrogen is usually deficient in mine soils and limits vegetation establishment and sustained productivity. Organic amendments are used as a source of mineralizable material to enhance N levels and extend N availability through cycling (Bradshaw, 1987).

Nitrogen is very mobile and easily leached in soil. Nitrogen undergoes many changes that are facilitated by soil biological, physical, and chemical processes (Brady and Weil, 1996). Total organic N added from organic amendments may be substantial to increase soil organic matter. However, a high percentage of potentially mineralizable N can be mineralized within the first year after application and continue to decline through time resulting in a rather short-term (1 to 5 yr) benefit to the N cycle (Barber, 1995).

The use of pig manure to reclaim mine soils in southeast Spain is promising because the region includes the province of Murcia where more than 10% of pig production in Spain is located. Annually, Murcia province generates an estimated 3.5 millions m<sup>3</sup> of waste residues from the pork industry (CAAMA, 2007). This generation of large volume of pig slurry continuously increases with high demands for pork, and consequently creates disposal problem for many pig producers. Earlier studies (Núñez-Delgado et al., 2002; Carmona et al., 2005) had shown that the organic matter content in pig manure (or dried slurry) can effectively increase soil organic matter and prevents migration of metals out of the soil. Using pig manure as soil amendment will address two environmental problems in southeast Spain – disposal of industrial wastes from pig production and reclamation of mine soils.

In this study, column leaching experiments were used to understand the dynamics of soil organic matter (SOM), particularly nitrogen in soils amended with pig manure to evaluate the potential use of organic waste residues for the remediation of mine soils.

## **MATERIAL and METHODS**

### **Undisturbed Column Mine Soil**

Three undisturbed soil columns were extracted using an apparatus designed based on Gavaskar (1999), Powell et al., (1998) and Park et al., (2002), and recent modifications by Carmona and Faz (2004) (Fig. 1A.). These columns were made of transparent methacrylate tubes (15-cm diameter and 60-cm length) with a base for plastic rings filter and an artificial rain system at the top. Soil columns were taken from surface to a depth of 35 cm of three representative soils affected by mining activities in the Mazarron District mining areas in Murcia, SE Spain.

### **Column Leaching Experiment**

The leaching column experiment was based on the studies by Mihaljevic et al., (2004), Ashworth and Alloway (2004), Doye and Duchesne (2003), Ciccu et al., (2003), Camobreco et al., (1996), and recommended by ISO/DIS 18772, (2006). Three columns were amended with single (3,750 kg N ha<sup>-1</sup> yr<sup>-1</sup>) and double doses of pig manure, incorporated with the mine soil in the first 10 cm of the column. The applied dose was obtained from the 3,580 kg N ha<sup>-1</sup> yr<sup>-1</sup> which is the mean nitrogen (N) content of soils in the study area, and the agronomic rate of N-requirement estimated at 170 kg N ha<sup>-1</sup> yr<sup>-1</sup> (Directiva 91/676/CEE). The pig manure amendment used in the study came from a farm located in Fuente Álamo-Murcia, and was taken from a dry pond.

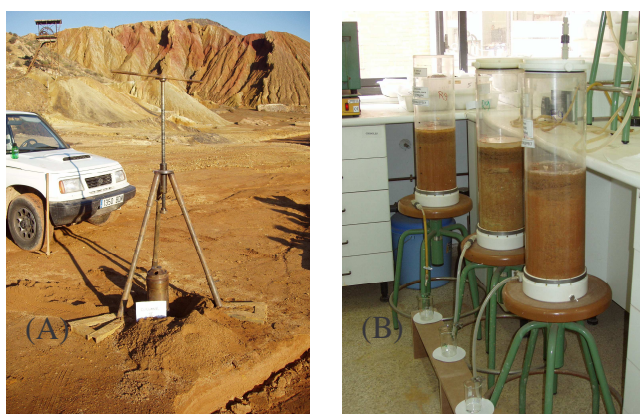


Figure 1. (A) Apparatus used to extract undisturbed soil columns. (B) Set-up of column leaching experiment.

Prior to leaching, mine soil column was saturated from the bottom using distilled water at a rate of 8 mL h<sup>-1</sup> applied using a peristaltic pump; the column remained saturated for 24 h to keep the oxygen content at minimum, silica sand was used as drainage material for efficient flow distribution. Leaching

was carried out weekly with 1,000 mL (50 L m<sup>-2</sup>) distilled water for 10 weeks to simulate annual critical rainfall events. At the end of week 7, we applied a 350 mm H<sub>2</sub>O corresponding to annual precipitation in the region. Experiment was carried out for 21 weeks (Fig. 1B.) for double doses. Leachates were collected as a function of time, for each week of experiment, and analyzed for pH, electrical conductivity (EC), redox potential (Eh), and soluble metals and ion contents. Results presented in this paper will be limited to total dissolved organic carbon (DOC), and nitrate (NO<sub>3</sub><sup>-</sup>) for leachates and total organic carbon (TOC) and total nitrogen for soils.

### **Analytical Methods**

Selected chemical properties of soils and pig manure were determined following the routine methods for soil and pig manure analyses. Soil pH and redox potential (Eh) were measured in 1:1 water extracts (Peech, 1965), electrical conductivity (EC) in 1:5 water extracts (Bower and Wilcox, 1965), Total organic carbon was measured using a TOC – SSM-5000A Shimadzu (Kyoto-Japan), and total nitrogen was determined by the Kjeldahl digestion-distillation method (Bremner, 1996). The chemical compositions of leachates were determined following analytical techniques suggested in APHA (1998), total DOC was measured using a TOC – V-CSH Shimadzu (Kyoto-Japan), and NO<sub>3</sub>–N by ion chromatograph (Dionex DX500-ED40) Soil columns leachates were filtered with a Whatman n° 42 paper, and filtrate were refrigerated at 5 °C while awaiting chemical analysis. We determined the following metals in soil and pig manure samples: Cd, Cu, Pb, Zn, and for leachates metals and ions were determined: Cd, Cu, Pb, Zn, iron (Fe), manganese (Mn), nitrate (NO<sub>3</sub><sup>-</sup>), sulphate (SO<sub>4</sub><sup>2-</sup>), chloride (Cl<sup>-</sup>), sodium (Na), potassium (K), calcium (Ca), and magnesium (Mg).

Statistical comparison of the chemical composition of soils and leachates from columns subjected to single and double doses of pig manure was conducted using ANOVA.

## **RESULTS and DISCUSSION**

Selected properties of pig manure and soils before and after the leaching experiment

Pig manure had higher pH, and content nitrogen and carbon, and lower EC, Eh (Table 1). Textural class of mine soil is silt loam, (FAO-ISRIC-ISSS, 1990), with a content of 5.9% clay; 29.3% fine silt; 30.8% coarse silt and 33.9% sand. The low pH of mine soil is often attributed to the oxidation of pyritic materials in mine wastes also known as acid mine drainage (Evangelou, 1995). Low content nitrogen and TOC indicate that mine soil are poor soil fertility and limit of grown plant. Addition of pig manure may help pH increase the low pH of mine soil. Similarly, the NO<sub>3</sub><sup>-</sup> in pig manure can potentially increase soil organic matter.

Table 1. pH, EC, Eh, N total, TOC (mean and standard error) in soils from column experiments.

	pH	EC dS m <sup>-1</sup>	Eh mV	Ntot g kg <sup>-1</sup>	TOC g kg <sup>-1</sup>	C/N
<b>Pig manure</b>	7.79	9.31	126	28.10	340.5	12.1
<b>Initial mine soil</b>	2.21 (0.07)	14.32 (2.25)	520 (30)	0.11 (0.03)	0.75 (0.17)	5.1
<b>W1</b>	5.99 (0.29)	2.57 (0.07)a	261 (35)	1.72 (0.29)	16.64 (0.35)	9.7
<b>W11</b>	3.98 (1.53)	1.78 (0.14)b	292 (136)	1.14 (0.24)	18.94 (6.90)	16.6
<b>W21</b>	6.34 (0.08)	1.30 (1.21)ba	173 (52)	1.40 (0.34)	16.65 (1.87)	11.9

n: number of observations in a group, w1 (n=3), w11 (n=3), w21 (n=2). Means followed by similar letter are not significantly differences

Amended soil columns showed that pig manure increased soil pH in single dose (W1 to W11) from 2.2 to around 4.0, 11 weeks after the addition of pig manure. The increase in soil pH was especially evident in soils amended with double doses where soil pH was 6.3 after week 21. The change in pH might indicate the “liming effect” of pig manure when added to the surface of mine soils. Soil EC values for both single and double doses progressively decreased from 14.3 to around 1.0 dS m<sup>-1</sup> perhaps, due to the removal of soluble salts from mine soil and pig manure through leaching. Eh was maintained at reducing conditions (290 and 170 mV, respectively for 11 and 21 weeks) (Figure. 1a). After addition of pig manure-amendment in top 10-cm of the column, significant increased were observed in total nitrogen contents in both single and double doses, 1.14 g kg<sup>-1</sup> (900%) and 1.40 g kg<sup>-1</sup> (1100%), respectively. Total carbon contents improved to 18.9 g kg<sup>-1</sup> (2400%) in single dose and to 16.6 g kg<sup>-1</sup> (2100%) in double doses. Nitrogen and carbon in soils had weekly rate of increases of 0.1 and 2.0 g kg<sup>-1</sup>, respectively. Moreover C/N ratio increased from 5 to 12 at the end of the experiment, this is quite significant because mine soil has almost negligible amount of N and TOC (0.11 and 0.75 g kg<sup>-1</sup> soil, respectively). Mine soil amended with pig manure can increase SOM and might initiate increase in microbiological population. Soil organic matter will also lead to increase in cation exchange capacity (CEC) and water holding capacity (Stevenson, 1994).

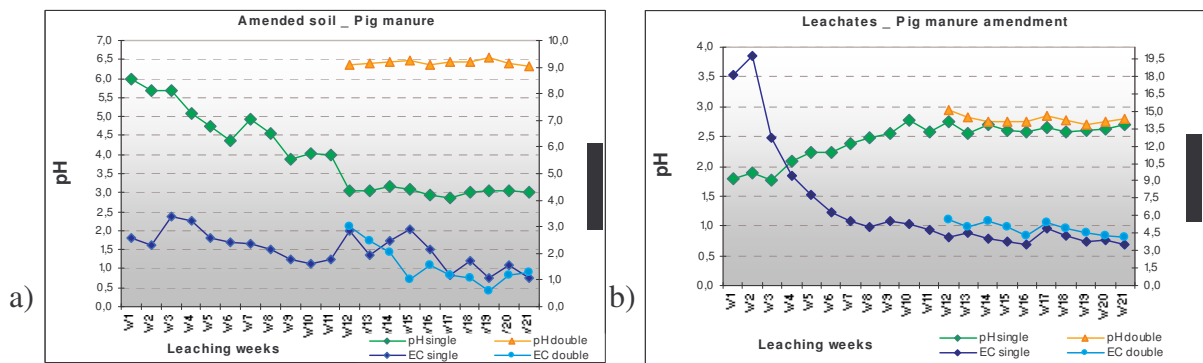


Figure 1. Dynamic changes of leachates pH and EC in the leaching experiment: (a) amended soil, (b) leachates

Leachates chemistry at the end of experiment (21 weeks)

The composition of leachates obtained in soil columns amended with pig manure subjected to single and double doses are presented in Table 2. The results showed that initial acidity was very high due to the oxidation of pyritic minerals as mentioned before. pH in leachates slightly increased from 1.8 to 2.6 after week 11 and to pH 2.9 at the end of the experiment. Leaching caused a significant reduction ( $p < 0.05$ ) in EC in single dose (W1-W11). EC values was highest in the first leaching events due to the washing of soluble salts from pig manure amendment and soil but declined from 18.1 to ~ 4.5 dS m<sup>-1</sup> in single and double doses (Fig. 1b). The redox potential in leachates remained in the range 400 to 450 mV for columns amended with single and double doses.

Table 2. Characteristics of leachates in single and double doses of pig manure-amendment

Leaching, week	pH	EC dS m <sup>-1</sup>	Eh mV	NO <sub>3</sub> <sup>-</sup> mg L <sup>-1</sup>	DOC mg L <sup>-1</sup>
W1	1.80 (0.08)a	18.13 (2.15)a	583 (24.9)a	42.1 (36.2)	25.25 (1.3)a
W11	2.57 (0.14)b	4.79 (0.33)b	412 (47)b	3.88 (1.24)	15.34 (1.6)b
W21	2.87 (0.19)ba	4.26 (0.49)b	448 (59)b	22.1 (27.4)	15.00 (2.9)ba

n: number of observations in a group, w1 (n=3), w11 (n=3), w21 (n=2). Means followed by similar letter are not significantly differences

Leaching of the nitrate and total DOC from the different doses of pig manure amendment is presented both as concentration in the leachate (Table 2) and as a rate of release (Fig. 2). Soil organic matter in this long-term experiment showed that significant changes had taken place during first 7 weeks, leachates had significantly higher weekly release of NO<sub>3</sub><sup>-</sup> than DOC during the first 7 weeks of leaching. Weekly rate of releases showed significant differences, the slopes founded were 127 mgNO<sub>3</sub><sup>-</sup> L<sup>-1</sup> (or 10.6 mg kg<sup>-1</sup>soil) and 5.2 mgDOC L<sup>-1</sup> (or 0.3 mg kg<sup>-1</sup>soil) in single dose, and 35 mgNO<sub>3</sub><sup>-</sup> L<sup>-1</sup> (or 2.1 mg kg<sup>-1</sup>soil) and 2.8 (mgDOC L<sup>-1</sup> (or 0.2 mg kg<sup>-1</sup>soil) for double doses.

This results suggest low losses of nitrate and DOC through soil washing processes especially at the initial application of pig manure. DOC concentration was highest in the first leachate and decreased during the experiment due to the removal of the most soluble dissolved organic matter (DOM) fractions. DOC release should be controlled by the decomposition of organic substances from pig manure. The increased in SOM of mine soil reduced the release of added NO<sub>3</sub><sup>-</sup> from the double doses addition perhaps due to increase microbiological activity that can consume the freshly-added NO<sub>3</sub><sup>-</sup>.

After 7 weeks, we estimated for single dose a total release of 74 mg NO<sub>3</sub><sup>-</sup> kg<sup>-1</sup> soil (17 mg N kg<sup>-1</sup>soil) and 15 mg NO<sub>3</sub><sup>-</sup> kg<sup>-1</sup> soil (3.3 mg N kg<sup>-1</sup>soil) for double dose, wich is very low compared to initial content in amended soil (1.7 g N kg<sup>-1</sup> soil).This result suggests that losses of N through leaching is very low and in a long-term (3 year) period, nitrogen content will stabilize at acceptable level that is conducive for plant growth.

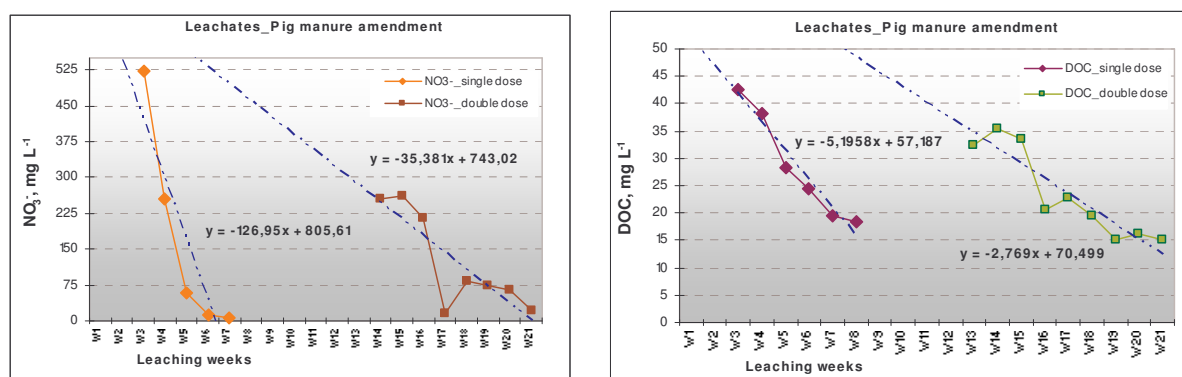


Figure 2. Rate release (mean) to  $\text{NO}_3^-$  and DOC in leachates for single and double doses.

In general, the changes in leachate  $\text{NO}_3^-$  concentration vs. leaching events had similar trends to those of DOC concentrations. However, there were evident differences between the contents for  $\text{NO}_3^-$  and DOC for both single and double doses. First, higher concentrations were noted for  $\text{NO}_3^-$  than the DOC, the maximum concentrations was obtained at week 5 given  $520 \text{ mg L}^{-1}$  for  $\text{NO}_3^-$  and  $43 \text{ mg L}^{-1}$  in single dose (W1-W11) (data not shown). Secondly,  $\text{NO}_3^-$  losses were relatively fast after pig manure application both in single and double doses;  $\text{NO}_3^-$  in leachates reached a minimum value ( $22 \text{ mg L}^{-1}$ ) at week 21. After week 6, leachates  $\text{NO}_3^-$  values were  $3.9$  and  $22.0 \text{ mg L}^{-1}$  for single and double doses, respectively. These  $\text{NO}_3^-$  contents remained less than the  $50 \text{ mg L}^{-1}$  threshold value established for irrigation water by FAO guideline (FAO, 1985). These results suggest that addition of pig manure may significantly accelerate the build up of SOM in mine soils without significant release of  $\text{NO}_3^-$  into sub-soil or groundwater in semiarid regions. Once there is sufficient SOM, mine soils will have an environment hospitable to various ecosystems including plant colonization and microbial community needed for its physical and chemical stability.

The long-term restoration of soil quality and permanent re-establishment of vegetation depends on the continuous N mineralization. Due to the decay in mineralizable N through time, the N content should be monitored especially on soils that have received a one-time application of pig manure. Marrs et al. (1983) explain that N accumulation is very slow in new ecosystems and that total soil N capital should be maintained at least 10 to 20 times the annual plant uptake.

## CONCLUSIONS

The results obtained from laboratory column experiment with additions of 7 % and 14 % (weight basis) of pig manure as amendment in mine soils show some potential benefits of pig manure as an agricultural amendment. Although, the high pH of pig manure was insufficient to neutralize the acidic pH in soil and leachates, pig manure amendment increased the total N, TOC and C:N ratio of mine soils. This is important because increases in SOM can initiate several important biochemical processes in soils. Increased soil organic matter can potentially increase soil pH, nitrogen content and

diminish metal mobility in mine soils. The low rates of NO<sub>3</sub><sup>-</sup> and DOC releases are additional findings to support the potential use of pig manure as an agricultural amendment to reclaim unproductive soils in several mining districts in southeast Spain. Our results suggest that mine soil amended with pig manure in double doses will not result to the migration through leaching of NO<sub>3</sub><sup>-</sup> to surface and ground water in semiarid conditions similar to the study areas.

Our results imply that using pig manure will help solve the problem of waste disposal simultaneously with the rehabilitation of unproductive soils in mined areas. The potential use of wastes from the ever growing large pig industry in Spain would help transform unhealthy soils left behind by more than 2,500 years history of mining in southeast Spain into land with functional ecosystems. Pig manure amendment of mine soils can be an ecologically-sound means of managing the large volume of wastes generated by the pig industry in southeast Spain. We propose that the use of pig manure as soil amendment is an environmentally-friendly approach to waste disposal at the same time might help reclaim long-time remediation program for mine soils.

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