



Article The Impact of 9 Years of Swine Wastewater Application on the Mineral and Organic Quality of Soil in Various Agricultural Crops

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Abstract: This study evaluates the long-term effects of swine wastewater (SWW) on relevant parameters for soil fertility, including calcium (Ca), magnesium (Mg), potassium (K) cations, cation exchange capacity (CEC), and organic matter (OM) in an agricultural area with 9 years of crop cultivation. Three types of SWW (raw, after leaving the biodigester, and after the manure plant) were analyzed with four application rates of SWW (0, 100, 200, and 300 m³.ha⁻¹), associated or not with mineral fertilization, resulting in eight treatments. The study found that the long-term use of SWW had significant effects on soil parameters. Principal component analysis (PCA) was used to summarize the data. The soil's calcium (Ca), magnesium (Mg), and cation exchange capacity (CEC) levels were higher in soybean compared to other crops and natural soil. Similarly, the treatment with 0 m³.ha⁻¹ of pig manure and without mineral fertilization showed higher levels of these nutrients. In contrast, potassium (K) was found in greater quantities in oats, SWW from the biodigester, higher doses of manure, and with mineral fertilization. The crops had a higher organic matter (OM) content compared to the natural soil, with corn and raw SWW showing the most significant increase.

Keywords: biodigester; mineral fertilization; nutrients

1. Introduction

Over the last five decades, Brazil has gained a leading position in the global agricultural commodities export market [1]. Against this backdrop, it is expected that by aligning with other countries in the sector, such as the United States, Russia, India, Canada, and China, and expanding exports to more than 175 countries, there will be advances in sustainability in the countryside [2].

The expansion of sustainable systems in the agricultural sector is a development strategy aimed at ensuring global food security and the long-term production of nutritious food [3]. To achieve this, it is essential to consider comprehensive sustainable soil and water management practices, including soil and nutrient management, balanced fertilizer use, and proper waste management [4].

Several studies have emerged globally, focusing on the treatment of swine wastewater (SWW) [5–7]. The increasing practice of livestock farming as a significant global economic source has led to the disposal and reuse of agricultural waste in water systems and soils [8,9]. The global concern over the presence of heavy metals, antibiotics, resistant pathogens, and nutrients from animal husbandry and care in SWW has been well documented in various countries [10]. In 2022, China alone produced the highest amount of pork at 55,000 tons, followed by the European Union (22,600 tons), the United States (12,200 tons), and Brazil (4300 tons) [11].



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Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Due to the high volume of wastewater produced on mainly industrial-scale farms, it is essential to reduce the nutrient load before applying it to the soil or discharging it into water bodies. Several studies have emphasized this need, including Li [12], Hao [13], Chen [14], Wang [15], Dai [16], and Tang [17]. The studies concluded that pig farm wastewater contains high concentrations of nitrogen and phosphorus, as well as organic matter, heavy metals, sulfides, and antibiotics. If disposed of directly, these substances can harm the environment and even human health. These studies have proposed various methods for treating and reducing the organic load, including the use of up-flow anaerobic sludge blanket (UASB), anaerobic membrane bioreactor (AnMBR), ecological bioflocculation, electrodialysis, and even the construction of wetlands with microalgae. However, the high cost of installing and operating these treatment systems, especially SWW, as well as the need for skilled labor, render the process viable only for large pig farms with limited agricultural areas for waste disposal [9].

The production model used in much of South America involves confining animals on small farms and using the soil as a destination for their waste, in contrast to extensive pig complexes. According to Deng et al. [9], this discharge is expected to increase with the global rise in pork production.

These farms utilize lagoons lined with blankets, also known as manure ponds, to store the animal waste. The waste is then applied to the soil as a method of disposal [18]. Additionally, there are pig farms that use biodigesters to produce biogas for electricity and heat. However, even with this technology, it is still necessary to dispose of the nutrient-rich digestate in the soil [19–21].

The potential of using SWW as a nutritional supply to agricultural areas for significant gains in crop productivity has shown promise in contributing to the goal of global food security, as well as ensuring nutrient cycling and reducing the use of mineral fertilizers [22,23].

Previous research has been conducted to evaluate the short- and long-term effects of swine manure and wastewater application on various soil and plant parameters, such as nutrient (e.g., N, P, K, Mg, Ca), heavy metal (e.g., Cu, Zn), organic matter (OM), cation exchange capacity (CEC), and pH concentrations [24–27]. However, although promising results have been obtained in terms of agronomic and chemical quality for short and medium-term applications (i.e., [26,27]), more attention needs to be paid to long-term applications, mainly due to the high concentration of heavy metals found in pig waste [28].

Organic soil amendment strategies are some of the alternatives commonly used to reduce the bioavailability of heavy metals, mainly due to their economic (i.e., low cost) and ecological (i.e., revegetation) advantages [29]. In SWW irrigation systems, studies have reported a reduction in Cu and Zn levels through the supply of OM resulting from the decomposition process of straw in contact with wastewater [24]. Immobilization is possible due to the complexation of metals with OM functional groups. However, it is essential to monitor the degradation rate and initial OM levels in the soil in which it is inserted, as the process can alter soil pH concentrations and promote the release of metals over time, as well as reducing CEC [29].

CEC represents the reversible binding capacity of polarized molecules and ions to electrically charged particles present in soils, thus allowing for ideal fertility conditions [30]. Due to the negative charges in the soil, high CEC values, together with greater adsorption, especially of the basic cations Ca^{2+} , Mg^{2+} , and K^+ (essential), are a good indication of nutritious soils, receptive to cations in their exchangeable form. In the case of correcting soil acidity, for example, it is recommended to use materials capable of providing anions that react with potentially toxic cations present in the soil (e.g., H^+ and Al^{3+}) to release the previously occupied anions present in the soil [30]. The Na⁺ cation is also considered an exchangeable base cation, but its presence in wetland soils such as those found in Brazil is rudimentary [31]. It is common in recent studies of pig manure fertilized soils to evaluate the effects of its application based on NPK content [24,32,33] due to the relevance of these macronutrients for productivity, especially N and P [34,35]. However, isolated analyses of Ca, Mg, and K mineral behavior are scarce.

For this reason, this study aimed to evaluate the long-term effects of applying different SWW in 24 drainage lysimeters, only for the three basic minerals (Ca, Mg, K) which, in their exchangeable form, play a direct role in the nutrient dynamics of wetland soils. Cation exchange capacity and organic matter were also determined for the treatments.

2. Materials and Methods

2.1. Characterization and History of the Experimental Area

The experimental area is situated at the Experimental Nucleus of Agricultural Engineering (NEEA) at the State University of Western Paraná in Cascavel, Paraná, Brazil (Figure 1). The geographical coordinates of the area are 24°54′2.30″ S 53°31′59.72″ W, and it has an altitude of 760 m.



Figure 1. Location of the experimental area at the Experimental Center for Agricultural Engineering (NEEA) in Cascavel, Paraná State, Brazil.

The region's climate is classified as humid subtropical according to the Köppen classification [36]. It is characterized by hot summers, an oceanic climate without a dry season, and rainfall of over 40 mm in the driest months with an average annual precipitation of 1800 mm, an average temperature of 20 °C, and relative air humidity of 73% [36]. The soil in the area is classified as typical dystrophic red latosol, which has a very clayey texture [37,38].

Experimental studies were conducted between 2006 and 2014, totaling nine years. For the long-term analysis of SWW application in soil with a no-till system for agricultural

crops commonly grown in the western region of Paraná, fifteen experiments with similar characteristics were selected.

2.2. Description of the Treatments

The study was conducted using twenty-four drainage lysimeters, each corresponding to an experimental plot of 1 m^3 in volume and 1.60 m^2 in area (0.91 m deep and 1.43 m in diameter), with a spacing of 0.4 m between them. Prior [39] provides a detailed description of the lysimeter construction.

Before their setup in 2006, a soil analysis was conducted to characterize the experimental area (SoloNat) (refer to Table 1). Soil analysis was conducted on each lysimeter after every crop harvest. Samples were taken from depths of 0–20 cm, 20–40 cm, and 40–60 cm, and then averaged [40].

Table 1. Chemical characterization of the soil in the experimental area (SoloNat) before the experiments were set up.

Blocks	pН	ОМ	Р	H + Al	K	Ca	Mg	SB ¹	CEC ²	V ³	Cu	Fe	Mn	Zn
	CaCl ₂	g.dm ⁻³	mg.dm ⁻³		mmolc.dm ⁻³			% mg.dm ⁻³						
B1	6.4	16.0	4.0	27.4	2.0	50.6	35.6	88.2	115.6	76.3	9.2	66.2	56.6	1.2
B2	5.1	15.0	4.0	46.1	1.8	30.0	15.9	47.7	93.8	50.8	8.7	64.4	35.8	0.8
B3	4.9	11.0	1.0	42.8	0.6	21.6	12.7	34.9	77.7	44.9	7.8	76.9	25.3	0.4
Average	5.47	14.0	3.0	38.8	1.5	34.1	21.4	56.9	95.7	57.3	8.6	69.2	39.2	0.8

Notes: ¹ SB—sum of bases; ² CEC—cation exchange capacity; ³ V = CEC/SB Source: Prior [39].

The SWW utilized in the experiments originated from a pig manure treatment system located on a rural property in the municipality of Toledo, PR. The manure was collected after leaving the manure house, after being removed from the biodigester, and before entering the biodigester (raw). Before application to the soil, each SWW collected underwent physical–chemical analysis for characterization in accordance with American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF) [41]. Table 2 displays the type of SWW utilized in each experiment.

Table 2. Characterization of the swine wastewater (SWW) from each experiment between 2006 and 2014.

Exp Local S	ww	Soy 2006	Soy 2007 Manure p	MiniMaize 2008 plant	Corn 2009	Oats 2009	Soy 2009	Soy 2010 Biodigest	Corn 2011 er	Oats 2011	Soy 2012	Corn 2012	Oats 2012	Corn 2013 Raw	Soy 2013	Corn 2014
pН		7.73	7.70	7.92	7.57	7.08	7.09	7.62	7.61	7.41	-	-	-	7.29	7.60	7.80
Nitrate		1.52	2.18	8.00	-	-	-	103.75	53.75	192.50	-	-	-	-	-	-
Nitrite		2.03	2.25	0.40	-	-	-	50.00	25.00	75.00	-	-	-	-	-	-
N		801	887	338	265	1278	604	481	351	975	707	105	980	707	2478	1050
Р		92.19	108.62	21.13	69.4	145.1	107.4	22.0	13.8	68.8	33.0	34.2	94.9	15.9	304.9	181.0
K		543	462	2.00	86	445	224	8.95	19.64	534	265	171	355	2.40	373	483
Ca	T	50.97	28.60	2.25	46.00	196.50	87.00	52.87	57.16	60.30	236.00	99.00	579.70	1.10	699.00	480.00
Mg	Ļ,	23.77	39.12	0.95	48.00	86.50	62.50	67.70	69.93	31.70	67.00	64.20	134.20	0.50	179.00	68.00
Na	ũ	18.20	26.00	1.00	79.20	166.70	125.00	36.70	35.40	143.00	16.80	68.00	140.00	-	20.80	167.00
Cu	н	0.20	0.25	12.50	0.72	5.05	1.96	1.86	1.80	0.90	8.30	0.50	28.10	1.00	6.27	3.70
Zn		1.17	0.20	76.50	6.50	35.00	14.50	10.22	11.30	3.56	39.00	6.32	181.50	4.00	5.71	4.70
TOC		-	-	-	40,500	2250	90,000	684	441	1077	29,160	530	9013	1988	2651	1988
TC		-	-	-	-	-	-	1331	822	1837	-	-	-	-	-	-
TIC		-	-	-	-	-	-	645	381	795	-	-	-	-	-	-

Notes: Protocol from APHA, AWWA, and WEF [41]. N: total nitrogen; P: phosphorus; K: potassium; Ca: calcium; Mg: magnesium; Na: sodium; Cu: copper; Zn: zinc; TOC: total organic carbon; TC: total carbon; TIC: total inorganic carbon.

The SWW application rates were defined and standardized during the first experiments at 0, 100, 200, and 300 m³.ha⁻¹, associated or not with mineral fertilization, resulting in eight treatments with three replications each (Table 3).

Treatments	SWW	Mineral Fertilization
1	$0 \text{ m}^3.\text{ha}^{-1}$	Without
2	$0 { m m}^3.{ m ha}^{-1}$	With
3	$100 \text{ m}^3.\text{ha}^{-1}$	Without
4	$100 \text{ m}^3.\text{ha}^{-1}$	With
5	$200 \text{ m}^3.\text{ha}^{-1}$	Without
6	$200 \text{ m}^3.\text{ha}^{-1}$	With
7	$300 \text{ m}^3.\text{ha}^{-1}$	Without
8	$300 \text{ m}^3.\text{ha}^{-1}$	With

Table 3. Treatments used in the experiments.

The swine wastewater (SWW) was manually applied using a watering can in just one step, seven days before sowing each crop. Cultural treatments (mineral fertilization and management of pests, diseases, and weeds) were the same for each harvest, following the needs of each crop, differing only between years.

The study assessed relevant parameters for soil fertility, including calcium (Ca), magnesium (Mg), potassium (K) cations, cation exchange capacity (CEC), and organic matter (OM).

2.3. Data Analysis

The soil characteristics measured at the end of each experiment, including K, Ca, Mg, and CTC, were analyzed using bivariate dispersion and Pearson's correlation calculation. Principal component analysis (PCA) was then conducted to summarize the composite variables, known as principal components (PC), and limit the dimensionality to be assessed. To reduce the influence of high point observations, all variables were square root transformed prior to the PCA. The PCs were retained and interpreted following the Kaiser–Guttman criterion [42,43], which requires eigenvalues greater than 1. The retained PCs were then correlated with the original variables to identify the level of association between each variable and each PC. A threshold of |r| > 0.70 was considered representative. Finally, the data were interpreted using analysis of variance—ANOVA for general linear models (GLM) [44].

The generalized linear model (GLM) considered CPs as the response variable and used the place where the SWW was obtained, the type of crop, and a factorial arrangement between the variables that made up the treatments: pig manure and mineral fertilization as predictor variables. The GLM was written in computer language.

PC_i ~ SWW + Crop + Pig manure + Mineral fertilization + Pig manure × Mineral fertilization

where

PC_i = i-th Principal Component retained for PCA interpretation;

SWW = categorical factor representing the places where the wastewater is obtained: manure, biodigester and raw water;

Crop = categorical factor representing the crops: corn, soy, oats, mini-maize, and natural soil;

Pig manure = numerical factor representing the levels of pig manure: 0, 100, 200, and $300 \text{ m}^3.\text{ha}^{-1}$;

Mineral fertilization = categorical factor representing mineral fertilization: with and without.

As OM was not measured in the 2007 soybean experiment, it was evaluated separately from the PCA to avoid losing information. The level of association between organic matter and PCs was assessed using Pearson's correlation, and the same GLM was used to infer possible sources of variation in this characteristic. All analyses and graphical representations were performed using RStudio 2023.06.0+421 software [45]. A significance level of 5% was adopted for all analyses.

3. Results

The table below shows the minimum, maximum, and average values for each soil element in the fifteen experiments analyzed (Table 4).

	K Ca		Mg	CEC	ОМ	
Variation Range –		g/dm ³				
Minimum	0.20	3.07	10.50	72.10	14.00	
Average	3.16	58.02	33.61	116.19	27.47	
Maximum	14.30	211.00	119.00	348.00	47.00	

Table 4. Minimum, maximum, and average values of each element analyzed in the soil.

The PCA summarized approximately 81% of the total data variability in the first two components. The first component (PC1) had an eigenvalue of 2.23, accounting for 56% of the variability, and was negatively correlated with Ca, Mg, and CEC. This component did not show any significant variability associated with the SWW ($F_{(2.335)} = 1.319$ and p = 0.2687) but was significant for crops ($F_{(3.335)} = 7.492$ and p < 0.0001), pig manure ($F_{(1.335)} = 5.413$ and p = 0.0206), and mineral fertilization ($F_{(1.335)} = 10.55$ and p = 0.0013), with the soybean crop being the most distinct in relation to the natural soil, as it had higher concentrations of these cations, followed by corn, oats, and mini-maize (Figure 2a). The concentrations of these cations were similar in the wastewater from the biodigester, raw water, and manure plant (Figure 2b). However, higher concentrations were observed in the absence of pig manure (Figure 2c) and in treatments without the addition of mineral fertilizer (Figure 2d). The *F* and *p* statistics indicate that mineral fertilization and crop were the most significant sources of variation in these soil characteristics.



Figure 2. Averages and 95% confidence intervals (bars) for the values of the first principal component (PC1) in relation to crops (**a**), swine wastewater—SWW (**b**), pig manure (**c**), and mineral fertilizer (**d**). Variables associated (Ca = calcium, Mg = magnesium, CEC = cation exchange capacity) with PC1 and their respective correlations are shown next to the direction of the association (red arrow).

The second main component (PC2) accounted for 25% of the variability, with an eigenvalue of 1.01, and was negatively associated with K. This component showed variability significantly associated with the SWW ($F_{(2.335)} = 5.812$ and p = 0.0033), pig manure ($F_{(1.335)} = 117.4$ and p < 0.0001), and mineral fertilization ($F_{(1.335)} = 67.85$ and p < 0.0001). Although there were higher values for K in the oat crop, the types of crops had a slight influence on this component (crop: $F_{(3.335)} = 2.377$ and p = 0.0698; Figure 3a). Higher concentrations of this cation occurred in the biodigester wastewater (Figure 3b), as well as in relation to the gradual increase in pig manure (Figure 3c) and the addition of mineral fertilizer (Figure 3d). It can be seen from the observed values of the *F* and *p* statistics that the most relevant sources of variation in this soil characteristic were pig manure and mineral fertilization.

OM did not show any significant associations with any of the main components retained from the PCA (PC1: r = -0.27; and PC2: r = -0.10), nor with the treatments carried out in the experiments (pig manure × mineral fertilization: $F_{(1.311)} = 0.022$ and p = 0.883; pig manure: $F_{(1.311)} = 2.618$ and p = 0.107; mineral fertilization: $F_{(1.311)} = 2.504$ and p = 0.115). However, the SWW ($F_{(2.311)} = 43.18$ and p < 0.0001) and the crops ($F_{(3.311)} = 15.85$ and p < 0.0001) showed significant effects on this soil component. It can be seen that all the crops had higher values than the natural soil before the experiments, with a marked difference for the corn crop (Figure 4a). In relation to the wastewater sources, the lowest average was observed in the biodigester and the highest in the raw water (Figure 4b).



Figure 3. Averages and 95% confidence intervals (bars) for the values of the second principal component (PC2) in relation to crops (**a**), swine wastewater—SWW (**b**), pig manure (**c**), and mineral fertilizer (**d**). The variable associated (K = potassium) with PC2 and its respective correlation is shown next to the direction of the association (red arrow).



Figure 4. Averages and 95% confidence intervals (bars) for organic matter values in relation to crops (**a**) and swine wastewater (**b**).

4. Discussion

SWWs exhibit high variability in nutrient concentrations due to factors such as water management for washing facilities, animal age, and feed composition [46,47]. Some authors have noted the significant variations in wastewater compounds, making it challenging to recommend them as agronomic fertilizers [48,49].

This study demonstrates a similar pattern to the one mentioned previously, where the composition of the SWW varied greatly between experiments. Additionally, the location where the SWW is collected can also affect the nutrient levels, as shown in Table 2. However, Figure 2b indicates that there was no significant difference in the amounts of Ca, Mg, and CEC in the soil between the sites where the SWW was obtained. However, Sarto et al. [46] have reported a higher concentration of Mg in the SWW at the biodigester inlet compared to the biodigester outlet and the storage lagoon outlet.

The soil's Ca, Mg, and CEC levels were higher in the soybean crop than in the other crops and the natural soil (see Figure 2a). Latosols typically have low chemical fertility compared to natural soil, but their composition can be improved through the use of fertilizers and soil correction, making them more productive [30].

Soils under tropical conditions have a greater number of negative charges on their colloids, which primarily adsorb cations. The sum of cations adhered to colloids represents the CEC, which depends on the type and quantity of clay and organic matter. It is important to fill most of the CEC with essential cations such as Ca, Mg, and K in balanced proportions to ensure adequate plant nutrition [50].

As monocotyledons, oats, maize, and millet are expected to extract nutrients from the soil similarly, as found in this study [51] these crops typically have lower levels of cell wall components that bind to Ca, reducing the absorption capacity of this element from the soil compared to dicotyledons like soybeans. As a result, they present lower Ca levels in the plant [52].

However, nutrients in the soil can interfere with each other's actions, either through inhibition or synergism. For example, K can negatively affect the absorption of Ca and Mg [50]. Previous research has shown that lower levels of Ca and Mg were found in the soil after a period of maize cultivation. Additionally, some crops showed no difference in the levels of these components, possibly due to the plants' absorption capacity or the adhesion of these nutrients to the soil's organic matter [46,53].

A higher amount of these same elements was observed in the treatments with $0 \text{ m}^3.\text{ha}^{-1}$ of pig manure (Figure 2c) and without mineral fertilization (Figure 2d), while an inverse relationship with K was observed (Figure 3c,d). Mg is typically absorbed less than Ca or K by plants due to its low natural availability in soils, the acidity of the environment, and its relationship with other elements. When there is a limited availability of Ca and K, Mg

absorption tends to increase. On the other hand, excessive fertilization with Ca and K can lead to Mg deficiency due to competitive inhibition [50].

Regarding K (Figure 3), the most significant differences were observed between the doses of SWW applied (Figure 3c) and the addition of mineral fertilizer (Figure 3d). This nutrient is present in high quantities in the SWW applied (Table 2) and is also one of the nutrients found in NPK. Previous studies have also reported higher K content in the treatment with cattle manure compared to the control [53,54]. As previously mentioned, Ca and Mg can interfere with the absorption of K, and vice versa [50].

However, although it is one of the main minerals required by plants in large quantities, its bioavailability varies between soil and plant, depending on the mineral composition of the soil, the levels and forms of K present, and the weathering rates [55]. K is highly mobile in soil and can be easily leached [48,53,54]. Previous studies have reported high amounts of K in the leachate from treatments with high doses of SWW [56,57]. On the other hand, K can also be fixed in interlayers of primary or secondary minerals in the soil, serving as reservoirs, with the fixation release dynamics controlled by the addition (i.e., fertilizers) or exhaustion (i.e., plant uptake) of the mineral in the soil solution (i.e., exchangeable K⁺), pH, CEC, and other environmental factors [58].

In all crops, a higher amount of OM was observed in the soil compared to the natural soil. OM contains almost all the macro- and micronutrients, plays a crucial role in maintaining their balance, and increases the soil's buffering power, thereby maintaining and improving the physical, chemical, and biological properties of the environment [50,59]. The previous crop's straw in a no-till system can affect the soil's physical properties, while humified OM can impact its chemical characteristics [50].

Additionally, soils with raw agricultural residue contain more OM than those with residue from biodigester digestate, likely due to the anaerobic digestion process that occurs within the biodigester [60]. The use of organic fertilizers, particularly digestate, can enhance the sequestration of atmospheric CO₂, resulting in the stabilization of soil OM [47]. Comin and colleagues [61] conducted a 10-year experiment on corn and black oats, testing three types of fertilizer (urea, liquid pig manure, and pig manure compost), and found that the soil had a greater amount of carbon with the manure compost compared to the liquid manure. However, both manures increased the soil's OM and CEC. Consequently, the mechanisms that maintain C in soils are contingent upon management practices [62].

Organic fertilizers are a viable alternative to industrialized fertilizers, with the goal of enhancing crop productivity [63]. They can also enhance the chemical and biological quality of the soil by increasing OM, boosting the microbial community, and augmenting the macro and micronutrient content in the soil [64].

The application of SWW to soil on small farms aims to dispose of waste that can cause environmental problems when not managed properly [5,65]. SWW can also serve as a biofertilizer, reducing costs by decreasing the use of conventional fertilizers and complementing soil fertilization based on the nutritional needs of each crop [66].

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