Monitoring the Dynamics in Nitrogen and Phosphorus Contents during Aerobic Digestion of Swine Slurry

Kyo-Suk Lee, Michael Egyir, Dong-Sung Lee, Hyun-Gyu Jung, Sang-Phil Lee, Jae E. Yang and Doug-Young Chung

1 Department of Bio-Environmental Chemistry, College of Agriculture and Life Sciences, Chungnam National University, Daejeon 34134, Korea; kyoosuk@cnu.ac.kr (K.-S.L.); miki.egyir@gmail.com (M.E.); jung659@hanmail.net (H.-G.J.)
2 Institute of Agriculture Science, Chungnam National University, Daejeon 34134, Korea
3 National Agricultural Cooperative Federation, Seoul 04517, Korea; oieomo@gmail.com
4 Kangwon Institute of Inclusive Technology, Kangwon National University, Chuncheon 24341, Korea; tlsbhd777@kangwon.ac.kr
5 Department of Biological Environment, College of Agriculture and Life Science, Kangwon National University, Chuncheon 24341, Korea
* Correspondence: yanggay@kangwon.ac.kr (J.E.Y.); dychung@cnu.ac.kr (D.-Y.C.); Tel.: +82-33-250-6446 (J.E.Y.); +82-42-821-6739 (D.-Y.C.)

Abstract: This study aimed to monitor the changes in the content and types of nitrogen (N) and phosphorus (P) in swine slurry (SS) across three aerobic digestion processes in public livestock recycling centers. In total, 148 swine slurry samples, including raw swine slurry from the three digestion cells, were collected from 37 livestock recycling centers around Korea from early April to mid-July 2020. The physical and chemical characteristics including the total N and P and various types of N and P of the samples were determined. The contents of organic N and P were calculated as the difference between the total and mineral components of N and P. The results showed that solid separation and aerobic digestion of SS significantly reduced the amount of the total N in the effluent by almost 70.1%, but only reduced the total P by 0.17%, resulting in a significant reduction of available N and a very high P:N ratio. This study confirmed that continuous application of composted liquid swine slurry based on N content may lead to the overapplication of P. Therefore, further research is needed to improve the N content and lower the high P:N ratios.

Keywords: swine slurry; nitrogen; phosphorus; aerobic digestion process; livestock waste recycling

1. Introduction

Owing to the continuous demand for pig products in the Korean market, swine production systems have become increasingly industrialized and intensive. Consequently, the swine population in South Korea has grown more than three-fold since 1983, from 3.6 million to 11.1 million in 2020 [1]. It has therefore become imperative to properly manage the quantum of large excreta churned out daily from the burgeoning swine industry. SS (generally a mixture of feces and urine and possibly a small proportion of feed), is an important source of macronutrients (such as nitrogen, phosphorous, and potassium) and micronutrients (such as copper and zinc), which can be utilized by plants [2]. The nutrient content in SS varies depending on the age of the pigs, the rations, the temperature, the methods used to collect and store manure, and the moisture content. The application rate of SS as a fertilizer can be recommended by requirement of N or P depending on plants or the residual amount of N and/or P determined by soil analysis results. However, the availability of N and P in SS can be determined by their forms which can be influenced by factors such as digestion processes, including temperature, pH, rate and time of aeration, and multivalent cation species reacting with orthophosphate in SS. N availability is a strong
determinant of plant growth and crop productivity. Plants use several forms of N such as nitrate, nitrite, and ammonium. Ammonium-N (NH$_4^+$-N), which is a preferable N source for the future when global levels of CO$_2$ are predicted to increase, is the predominant inorganic form of N in SS, and it is immediately available to the crop following application. Nitrite availability can be determined by the balance between nitrification and denitrification, although its concentration in SS is generally lower than that of nitrate and ammonium [3]. The P content of the SS is highly variable. Mineralization of organic P to inorganic P contributes to the supply of plant available P. However, repeated applications of SS can result in soil P levels well above agronomic requirements that can be a serious contaminant at elevated levels in surface water. Therefore, a proper treatment of SS becomes essential to avoid the risks of contamination of water, soil, and air and to enhance N efficiency caused not only by N loss during the digestion process [4] but also by abysmally high P to N ratios [2,4].

In Korea, the majority of SS is processed in government-approved public livestock waste recycling centers (PLRC). These facilities adopt a treatment system comprising three-stage aeration cells with continuous or intermittent aeration. This is in accordance with livestock waste guidelines of the Ministry of Environment (ME) and the Ministry of Agriculture, Food, and Rural Affairs (MAFRA). The processed SS is applied as composted liquid swine slurry (CLSS) in the fields [5,6]. Mechanical separation of the total solids (TS) from raw swine slurry (RSS) is the first step in the SS management facility, which relies on the partitioning of slurry in a liquid stream [7]. The lower TS concentration in the separated liquid fraction from RSS reduces NH$_3$ emissions during the application stage due to a more rapid infiltration in soil [8]. The separation of solids from RSS influences the characteristics and contents of N and P in the resulting liquid [9]. Therefore, RSS must be separated into solid and liquid fractions before it can be treated and utilized.

Several studies have revealed that the continuous or intermittent aeration of the SS significantly reduced the nitrogen content in the final CLSS as a result of the nitrification and denitrification processes which evolves the nitrogen into gaseous forms such as NH$_3$ and N$_2$O [10,11]. Ammonium will convert to ammonia at a pH that is greater than 6.5. Increasing the pH (more alkaline or less acid) increases the amount of ammonia and decreases the amount of ammonium. Most manure of which a pH is close to 7.0 or higher contains both ammonium and ammonia [12]. This phenomenon mostly results in producing CLSS with a very high phosphorus to nitrogen ratio. Therefore, if an application rate of CLSS is based solely on its N content, it will result in overapplication of P which can have a deleterious impact on the environment as well as its accumulation in the soil. However, obtaining an accurate measurement of NH$_3$ volatilization from animal manure composting facilities is difficult and expensive because emissions are affected by differences in digestion conditions (aeration, pH, temperature, and environmental and management processes) [13–15].

Notwithstanding, monitoring the fractions and contents of N and P in CLSS during the composting period would be helpful in providing an insight into the suitable end-point for the aeration process so as to minimize the high P to N in the final product. However, few attempts have been made to monitor the nutrient components in CLSS produced from the PLRCs. Therefore, the aim of this study was to monitor changes in the content and forms of N and P in CLSS in the three stages of aerobic digestion in PLRCs, so as to come up with an appropriate treatment method and the time required in producing CLSS with a reduction of N loss, so as to minimize the negative environmental impacts associated with the overapplication of phosphorus. The results of this study may be useful for facilitating the implementation of treatment processes for SS and improving the availability of N in CLSS.

2. Materials and Methods

2.1. Sample Collection

Swine slurry from the storage tank of individual swine farms employing intensive farming for breeding and fattening was collected in a tank with a mechanically stirred
mixture. The SS delivered to the PLRC was pumped to the storage tank for the separation of solid substances, including impurities.

The guidelines of the MAFRA [16] dictate the specific information related to the facility design and digestion processes for fresh swine slurry (FSS) through three-stage aeration digestion cells for 90 days after removal of solids greater than 2 mm in diameter from RSS. As shown in Figure 1, the typical SS treatment system in PLRCs consisted of three consecutive equal, independent, isolated digestion cells. PLRCs first separate the solid substances from RSS using a screw press separator with a screen or a decanting centrifuge separator. Generally, the MAFRA does not recommend any chemicals for the efficient separation of solid substances and soluble P in the storage cell and/or pH adjustment over the three aeration cells during the digestion process. However, a few PLRCs used chemicals to neutralize the pH of the FSS when the pH of the RSS after separation of solid substances was higher than 8.5 and to improve the efficiency of the separation of solid substances before the centrifugal separation in the storage cell of RSS.

![Figure 1. Schematic illustration of typical swine slurry treatment scheme showing storage cell to maturation cell with air blowing unit (left) and picture of presently operating PLRC (right).](image)

The separation step of RSS included a screw press with 2.0 mm screen openings. Most PLRCs underwent a second separation step in a decanter centrifuge for the produced liquid fraction from the screw and then pumped to the first cell. Then, the separated SS is kept for a maximum of 30 days during each stage of the digestion process from FSS to maturation. Air is supplied to the different cells for different durations on daily basis, 20 h for the first cell, 10 h for the second cell and 2 h for the third cell. The rate and time of aeration for each cell could be adjusted by the treatment volume and biological oxygen demand as recommended by the MAFRA.

RSS samples were collected from the storage cell of the PLRCs, delivered from swine farms before mechanical separation, FSS samples were collected on the first day from the first aeration cell, immediately after the removal of solid particles greater than 2 mm in diameter from RSS via filtration. For the interim SS (I30, I60), I30 denotes samples collected on the first day from the second aeration cell, transferred from the first aeration cell after a 30 day digestion period of FSS, I60 denotes samples collected on the first day from the third aeration cell, transferred from the second aeration cell after a 30 day digestion period of I30. MSS denotes samples collected from the third aeration cell after a 30 day digestion period of I60.

The experiment was conducted by collecting samples from 37 PLRCs across the country and grouped into four categories based on the stage of the digestion process. In all, 148 samples were collected using bailer samplers from early April to mid-July 2020. Five-liter samples were collected from three depths at five different points in each cell for each sampling period, sealed inside polyethylene containers, and transported in a portable refrigerator at 4 °C to the laboratory for analysis within 24 h.

2.2. Sample Analysis

Samples were homogenized adhering to the APHA Standard Methods [17], by stirring for 10 min. The pH and electrical conductivity (EC) of the samples were immediately mea-
Table 1. Distribution and descriptive statistics of physical and chemical properties of RSS collected from 37 public livestock waste recycling centers.

| Category | TS | WC | OM | Ash | pH | EC | TN | TP |
|----------|----|----|----|-----|----|----|-----|----|
|          | (%) | (%) | (%) | (%) |    | (%) | (%) | (%) |
|          | Pr | Pr | Pr | Pr | (%) | (%) | (%) | (%) |
|          | Range | Range | Range | Range | STD | Range | Range | Range |
|          | Range | Range | Range | Range | (%) | Range | Range | Range |
|          | (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) |
|          | (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) |
| Distribution | 0-10 | 0.28 | 80-85 | 0.21 | 0.0-0.3 | 2.79 | 0.0-1.0 | 46.2 | 5.0-5.5 | 0.08 | 5-10 | 0.75 | 500-1000 | 0.28 | 0-250 | 0.28 |
|          | 1.0-2.5 | 1.23 | 85-90 | 1.49 | 0.5-1.0 | 4.13 | 1.5-2.5 | 46.8 | 5.5-6.0 | 3.32 | 15-15 | 1.46 | 300-2000 | 1.64 | 250-500 | 0.29 |
|          | 2.5-5.0 | 72.50 | 90-95 | 25.38 | 1.0-2.3 | 6.75 | 2.5-5.0 | 3.31 | 6.0-6.5 | 4.96 | 15-20 | 5.21 | 2000-3000 | 16.9 | 500-750 | 3.98 |
|          | 5.0-7.5 | 8.72 | 95-100 | 62.5 | 2.5-5.0 | 20.8 | 5.0-7.5 | 1.41 | 6.5-7.0 | 23.5 | 25-30 | 46.7 | 4000-5000 | 27.5 | 1000-1500 | 13.1 |
|          | 7.5-10 | 3.15 | 98-100 | 90.0 | 0.0-0.5 | 2.75 | 7.5-10 | 0.28 | 7.0-7.5 | 7.0-7.5 | 23.5 | 25-30 | 46.7 | 4000-5000 | 27.5 | 1000-1500 | 13.1 |
|          | 10-15 | 1.30 | 90-95 | 90.0 | 0.0-0.05 | 8.31 | 10-15 | 1.30 | 7.5-10 | 7.5-10 | 1.05 | 15-20 | 33.2 | 3000-4000 | 33.2 | 1500-2000 | 63.8 |
|          | 15-20 | 0.28 | 90-95 | 90.0 | 0.0-1.0 | 5.52 | 15-20 | 0.28 | 7.5-10 | 7.5-10 | 0.75 | 25-30 | 33.2 | 3000-4000 | 33.2 | 1500-2000 | 63.8 |
| Min.    | 0.24 | 90.0 | 90.0 | 0.0-1.0 | 0.58 | 0.05-0.75 | 0.12 | 0.0-0.1 | 0.12 | 0.05-0.1 | 1.34 | 0.05-0.1 | 1.34 | 0.05-0.1 | 1.34 | 0.05-0.1 | 1.34 |
| Max.    | 16.5 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 | 99.7 |
| SD      | 7.22 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 | 6.71 |

Table 2. Distribution and descriptive statistics of physical and chemical properties of FSS after separation of solid substances from RSS for 37 public livestock waste recycling centers.

| Category | TS | WC | OM | Ash | pH | EC | TN | TP |
|----------|----|----|----|-----|----|----|-----|----|
|          | (%) | (%) | (%) | (%) |    | (%) | (%) | (%) |
|          | Pr | Pr | Pr | Pr | (%) | (%) | (%) | (%) |
|          | Range | Range | Range | Range | STD | Range | Range | Range |
|          | Range | Range | Range | Range | (%) | Range | Range | Range |
|          | (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) |
| Distribution | 0-1 | 0.98 | 80-85 | 0.07 | 0.0-0.25 | 2.69 | 0.0-0.05 | 8.31 | 5.5-4.0 | 0.09 | 5-10 | 0.75 | 500-1000 | 0.75 | 0-250 | 0.75 |
|          | 1-2 | 0.91 | 85-90 | 0.08 | 0.25-0.15 | 7.21 | 0.05-0.11 | 53.2 | 6.0-4.5 | 4.04 | 10-15 | 3.69 | 1000-2000 | 4.24 | 250-500 | 3.52 |
|          | 2-3 | 7.93 | 90-95 | 0.15 | 0.5-0.75 | 10.0 | 0.1-0.15 | 27.4 | 6.5-7.0 | 10.5 | 15-20 | 3.69 | 2000-3000 | 45.8 | 500-750 | 20.8 |
|          | 3-4 | 0.99 | 95-100 | 0.27 | 0.75-1.0 | 12.6 | 0.15-0.2 | 11.1 | 7.0-7.5 | 30.6 | 20-25 | 45.8 | 5000-6000 | 41.5 | 750-1000 | 44.2 |
|          | 10-12 | 40.2 | 0.25-1.5 | 10.5 | 0.5-1.0 | 4.04 | 10-15 | 3.69 | 15-20 | 3.69 | 15-20 | 3.69 | 2000-3000 | 45.8 | 500-750 | 20.8 |
|          | 12.5-15 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 | 10.5 |
| Min.    | 0.04 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 | 90.0 |
| Max.    | 5.28 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 | 99.8 |
| SD      | 0.78 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 |

TS, total solid; WC, water content; OM, organic matter content; EC, electrical conductivity; TN, total nitrogen; TP, total phosphorus; SD, standard deviation; CV, coefficient of variance; Pr, proportion.

To determine the cation species including heavy metals and contents, 10 mL of the FSS sample was first digested with concentrated nitric acid (V) [17]. Subsequently, the solution was made up to 100 mL in a volumetric flask and quantitatively analyzed using inductively
coupled plasma (ICP) atomic emission spectroscopy (AES) (ICPS-7510, Shimadzu Corp., Kyoto, Japan) (Table 3).

**Table 3.** Content of various cations and heavy metal in fresh swine slurry.

| Cation | Raw (mg L\(^{-1}\)) |
|--------|---------------------|
| Na     | Max: 659.7 2919      |
|        | Min: 30.1 195.5      |
|        | Median: 338.8 245.5  |
|        | Mean: 324.6 591.1    |
| K      | Max: 255.5 255.8     |
|        | Min: 21.4 3.48       |
|        | Median: 118.8 158.3  |
|        | Mean: 132.4 159.6    |
| Ca     | Max: 316.3 302.2     |
|        | Min: 61.3 63.2       |
|        | Median: 184.4 107.3  |
|        | Mean: 129.9 124.6    |
| Mg     | Max: 302.2 386.1     |
|        | Min: 63.2 107.9      |
|        | Median: 107.9 107.9  |
|        | Mean: 107.3 107.9    |
| Fe     | Max: 316.3 302.2     |
|        | Min: 63.2 107.9      |
|        | Median: 107.9 107.9  |
|        | Mean: 107.3 107.9    |
| Cu     | Max: 44.8% 48.8%     |
|        | Min: 1.0–2.5% 1.0–2.5%|
|        | Median: 7.5–8.0%    |
| Zn     | Max: 146.1 124.6     |
|        | Min: 0.05–0.1%       |
|        | Median: 1.16 1.16    |
| As     | Max: 0.21 0.04       |
|        | Min: 0.003 0.003     |
|        | Median: 0.04 0.04    |
| Cd     | Max: 2.10 1.35       |
|        | Min: 0.01 0.05       |
|        | Median: 1.0 0.88     |
| Cr     | Max: 1.90 0.78       |
|        | Min: 0.04 0.05       |
|        | Median: 1.7 0.88     |
| Ni     | Max: 2.44 3.07       |
|        | Min: 0.05 0.17       |
| Pb     | Max: 3.3 0.04        |
|        | Min: 0.05 0.05       |
|        | Median: 3.58 0.88    |

SD, standard deviation; CV, coefficient of variance; ND, not detected.

2.3. Statistical Analysis

Microsoft Office Excel 2019 (Washington, DC, USA) was used to analyze the experimental data and for graphing purposes (regression equations). Box plots were constructed to provide a visual summary of the distribution of physico-chemical properties including TN and TP, different species of nitrogen and phosphorus, and cations including heavy metals. For each measurement for the samples collected from 37 PLRCs, the mean, median, standard deviation (SD), and coefficient of variance (CV) were calculated using Microsoft Excel 2019. Pearson product–moment correlation coefficients were calculated by choosing the bivariate with the two-tailed option using SPSS (IBM SPSS Statistics version 24; IBM Corp., Armonk, NY, USA) to show the relationship between the slurry parameters.

3. Results and Discussion

All results from chemical and physical analyses performed on the samples from RSS and FSS are presented in Tables 1 and 2. The data from each measurement for RSS significantly varied because the SS generated in a swine farm depends on the type and the age of the pigs, their feeding system and maintenance, the quantity and quality of fodder, as well as the dilution of the slurry and its storage method [9,12,13]. The proportions of dominant ranges of each physical measurement of RSS were 72.3% (2.5–5.0%), 62.5% (95–100%), 67.5% (1.0–2.5%), and 48.8% (1.0–2.5%) for TS, WC, OM, and ash, respectively, while those of the chemical properties were 41.1% (7.5–8.0), 46.7% (25–30 dS m\(^{-1}\)), 37.5% (4000–5000 mg L\(^{-1}\)), and 63.8% (1500–2000 mg L\(^{-1}\)) for pH, EC, TN, and TP, respectively.

The largest difference between the maximum and minimum was in WC (18.6%), followed by TS (15.9%), ash (8.67%), and OM (7.35%), while the differences between the maximum and minimum of pH and EC were 3.05 and 35.8 dS m\(^{-1}\), respectively. The differences in TN and TP were 4911 mg L\(^{-1}\) and 1884 mg L\(^{-1}\), respectively (Table 1). This could be caused by high variability in swine diets, housing and cleaning systems, and the length of time depending on the type of storage for SS before it was delivered to the PLRCs from the swine farms.

For each measurement of FSS after separation of solid substances from RSS, the proportions of dominant ranges of TS, OM, and ash were 90.1% (1.0–2.0%), 40.3% (1.0–1.25%), and 53.2% (0.05–0.1%), respectively, while those of EC, TN, and TP were 45.8% (20–25 dS m\(^{-1}\)), 45.8% (2000–3000 mg L\(^{-1}\)), and 44.2% (750–1000 mg L\(^{-1}\)), respectively. It was evident from the results that the physical and chemical properties of FSS were lower than those of RSS except for WC. The differences of FSS between the maximum and the minimum for each measurement except EC were drastically decreased to 3.04% (TS of FSS), or below, respectively (Table 2). This could be caused by the decrease in solid substances, including OM, in FSS as a result of the separation of solid particles (>2 mm in diameter) after flocculation of suspended substances from RSS. The distribution of pH showed that the proportion of
pH greater than 7.5 was approximately 58.6%, indicating that NH$_4^+$ can be readily lost to the air by volatilization because NH$_4^+$ in solution can be easily transformed into NH$_3$ in pH greater than 7.5 although Org-N, which is the most abundant form of nitrogen in RSS, cannot be transformed into ammonia immediately [12].

Comparison of the mean contents of physical and chemical properties between RSS and FSS showed that each measurement of FSS was lower than those of RSS except WC and pH (Table 4). The decreased amounts of TN and TP of FSS were 759 mg L$^{-1}$ and 188 mg L$^{-1}$, corresponding to 26.1% and 16.3% of those of TN and TP of RSS, and 6.72% and 1.67% of OM decreased between RSS and FSS. The NH$_4^+$ form cannot be removed by the separation process and very little nitrate nitrogen is present in RSS [12]. Gilkinson and Frost [23] found that the decrease of TN content was positively correlated with OM after the separation of solids by the decanting centrifuge or the brushed screen. Therefore, we could assume that the significant loss of TN and TP between RSS and FSS was attributed to the separation of solid substances, including OM, from the RSS.

**Table 4.** Mean differences of physical and chemical properties between RSS and FSS.

| TS     | WC  | OM  | Ash | pH  | EC  | TN  | TP  |
|--------|-----|-----|-----|-----|-----|-----|-----|
| 2.36   | −2.1| 1.13| 1.9 | −0.03| 1.2 | 759 | 188.2|

TS, total solid; WC, water content; OM, organic matter content; EC, electrical conductivity; TN, total nitrogen; TP, total phosphorus.

In Table 3, we quantitatively determined the contents and species of cations, including heavy metals and anions in FSS, after the separation of solid particles from RSS. Great variation in the content of cations was observed in the samples. The most dominant cations were potassium (K), and Cu and Zn were the two dominant heavy metals in the FSS; As, Ni, Pb, Cd, and Cr were found in lower concentrations. It is important to note that metal accumulation in the soil is highly dependent on the nature of the slurry as well as on its application rate; multivalent cations can react with phosphate ions in the solution to make ionic crystals that precipitate, depending on the solution pH. Therefore, the amount of multivalent cations and the solution pH can determine the amount of phosphate during the digestion process.

Figures 2 and 3 show the changes of physical and chemical properties from RSS to MSS. The decreases in mean content of TS, OM, and ash between RSS and FSS were 2.36%, 1.13%, and 1.87%, respectively. Then those of TS and OM gradually declined to 1.29% and 0.23% in MSS (except ash), showing that the decreases in TS, OM, and ash between FSS and MSS were 0.30%, 0.52%, and less than 0.01%, respectively. However, WC was increased from 96.5% in RSS to 98.9% in MSS. Comparison of the changes for each measurement showed that the decreases in TS, OM, and ash between FSS and MSS were 11.3%, 31.5%, and <0.01% for the decreases in TS, OM, and ash between RSS and MSS (Figure 2). It can be interpreted that the decreases in these measurements were significantly influenced by the separation of solid substances from RSS, while the relatively higher reduction of OM could be attributed to the process being hastened by the aeration and microbial activity between FSS and MSS. The change of each measurement showed that it could be significantly influenced by the separation of solid substances from RSS, while the relatively higher reduction of OM between FSS and MSS could be attributed to the digestion process being hastened by the aeration and microbial activity over the three aeration cells after the separation of solid substances from RSS.
The mean pH was gradually increased from 7.75 in FSS to 8.19 in MSS, but the increase in pH was slowly retarded as the digestion processes over the three cells proceeded with different aeration rates and duration on a daily basis from FSS to MSS. The mean EC changed from 23.3 dS m$^{-1}$ in RSS to 23.9 dS m$^{-1}$ in MSS, respectively, while EC between RSS and FSS was decreased by 1.1 dS m$^{-1}$ that of RSS. Khan and Mostafa [24] observed that EC generally increases with the increase of total dissolved solids in SS. The separation of solid substances from RSS decreased the EC in FSS because of the increase of WC in the FSS by 2.1%. Similarly, the increases in pH and EC could be influenced by the increased surface charge from the increase in the smaller fraction of OM to buffer H$^+$ in the solution and the ionic species released in their soluble phase accompanied by the decomposition of OM [12]. For changes of pH as observed by Zhu et al. [25] and García-Gonzalez [26], an increase in pH by 0.44 unit during the aeration process between FSS and MSS may enhance the transformation of NH$_4^+$ into its gaseous form as NH$_3$.

The mean content of TN was changed from 2908 mg L$^{-1}$ to 886.3 mg L$^{-1}$ between RSS and MSS, with reductions in TN by 759 mg L$^{-1}$ between RSS and FSS (Figure 3). The reductions in TN and TP between RSS and FSS corresponded to 37.5% and 85.8% of the reductions between RSS and MSS. Approximately 75% of the TN, which occurs in organic and inorganic forms in SS, is ammonia, ammonium, nitrates, and nitric oxides [27–29]. Makara and Kowalski [9] reported that the separation of solid substances after chemical treatment, such as flocculants, results in the removal of solid constituents from the liquid.
fraction with the following removal rates: 40% of total Kjeldahl nitrogen (TKN) and 34% of TP.

Bicudo and Svoboda [30] reported that the aeration during the treatment of SS resulted in almost complete removal of soluble biodegradable matter and up to 95% removal of TKN with a very low ammonium level in the final effluent. Aeration of SS involves a huge loss of the valuable nutrient, N, due to the enhanced emission of NH$_3$ and N$_2$O by decreasing the amount of organic matter [31]. Among the components of TN and TP, N and P remained as the soluble phase in SS cannot be separated by mechanical separation such as a screw press separator with a screen or a decanting centrifuge separator used in PLRCs. Therefore, the form of TN reduced between RSS and FSS could be organically bound N while the form of TN reduced between FSS and MSS could be inorganic N such as NH$_3$ and N$_2$O because a major fraction of the organic N is rapidly hydrolyzed into its inorganic form such as NH$_4^+$ by the enzyme urinase which is present in the SS. Following which, the NH$_4^+$ can be transformed into NH$_3$ in pH greater than 7.0 and into nitrate by aeration during the digestion process of SS [32]. Nitrate produced by nitrification can remain in the solution while NH$_3$ may be lost from SS due to volatilization. Therefore, the reduction of TN can be attributed to organic N and inorganic N between RSS and FSS, and FSS and MSS, respectively. Approximately 70% of the N in SS is dissolved and most of the dissolved N is present as NH$_4^+$ [33]. Hence, the reduction of TN as NH$_3$ from FSS to MSS can be increased by increasing the pH of SS, as was observed in Figure 3.

Most of the P in SS is in the particle fraction of slurry, and 30% or more is dissolved in the liquid phase. An amount of 80% or more of the dissolved P is orthophosphate [7]. The mean content of TP was changed from 2012 mg L$^{-1}$ to 933.6 mg L$^{-1}$ between RSS and MSS, with reductions in TP by 188.2 mg L$^{-1}$ between RSS and FSS (Figure 3). The reductions in TP between RSS and FSS corresponded to 85.8% of the reduction between RSS and MSS. The reduction of TP after the separation of solid substances from RSS was more than twice as much as Makara and Kowalski’s result [9]. The most pronounced phenomenon of aeration is perhaps the pH rise in the SS. Aeration may increase the pH of the SS by approximately one unit within 24 h, from 6.5 to 7.5, and slowly increased the pH to 8.5 thereafter [34]. They also found that approximately 75% of the soluble P removal was achieved within the first day for aeration schemes, but further reduction was not observed in later days. This result suggests that the removal of P by aeration may be most effective in the first day of operation, and further aeration may not be needed in this regard. As seen in Figure 3, the pH rise in SS was retarded as the digestion processes moved from FSS to MSS because the rate and duration of intermittent aeration were decreased with the digestion stage. Therefore, we could assume that Org-P was dominant for the reduction of TP between RSS and FSS while soluble-P was dominant after FSS.

Table 5 shows the changes of content for different forms of N and P over three stages of aerobic digestion from FSS to MSS, including RSS. Air was supplied to the cells on a daily basis such as 20 h for the first cell, 10 h for the second cell, and 2 h for the third cell in PLRCs. During the aeration of SS, the anoxic and reductive conditions in which SS were stored change to aerobic and oxidizing conditions. However, nitrification and denitrification processes could occur during continuous or intermittent aeration in the digestion cell, producing nitrogen gases. Research on aerobic treatment of SS normally considers a longer duration of the aerobic phase in comparison to the anaerobic phase, and results indicate that nitrification and denitrification is enhanced [35]. However, aerobic treatment systems are not considered to be the best available techniques because they involve a loss of a valuable nutrient and the side effects of emissions such as N$_2$O and N$_2$ are still considerable [36].
Table 5. Variation in content of N and P in swine slurry samples from each stage of digestion.

| Types of SS | N          | P          |
|-------------|------------|------------|
|              | T-N        | Org-N      | NO$_3^-$ | NH$_4^+$ | T-P | Org-P | PO$_4^{3-}$ |
|              | (mg kg$^{-1}$) | (mg kg$^{-1}$) |          |          |     |        |             |
| RSS         | Max. 5873   | 2596       | 88.3     | 3276     | 2012 | 855.6  | 1165       |
|             | Min. 962.3  | 477.6      | 20.6     | 434.7    | 127.5 | 47.8   | 91.4        |
|             | Median 2857 | 1253       | 50.5     | 1618     | 1148 | 472.5  | 663.1       |
|             | Mean 2908   | 1285       | 49.8     | 1641     | 1153 | 479.6  | 665.9       |
|             | SD 2029     | 878.6      | 27.8     | 1167     | 770.8 | 330.1  | 438.8       |
|             | CV 0.70     | 0.68       | 0.56     | 0.71     | 0.67  | 0.69   | 0.66        |
| FSS         | Max. 4298   | 1124       | 85.2     | 3157     | 1703 | 541.5  | 1169       |
|             | Min. 689.2  | 258.1      | 19.1     | 424.3    | 114.5 | 35.1   | 92.6        |
|             | Median 2111 | 527.6      | 49.3     | 1569     | 957.6 | 311.5  | 666.4       |
|             | Mean 2149   | 533.4      | 48.9     | 1578     | 964.8 | 312.8  | 668.4       |
|             | SD 1797     | 365.5      | 27.0     | 1123     | 649.2 | 207.2  | 439.9       |
|             | CV 0.67     | 0.69       | 0.55     | 0.71     | 0.67  | 0.66   | 0.66        |
| I30         | Max. 3148   | 738        | 295.5    | 2099     | 1689 | 521.6  | 1161       |
|             | Min. 455.4  | 165.2      | 43.8     | 233.1    | 108.4 | 22.7   | 97.4        |
|             | Median 1625 | 337.7      | 228.5    | 1038     | 948.9 | 292.4  | 680.2       |
|             | Mean 1654   | 341.4      | 230.4    | 1041     | 949.8 | 291.3  | 681.6       |
|             | SD 1103     | 242.8      | 108.4    | 765.2    | 645.9 | 204.0  | 435.2       |
|             | CV 0.67     | 0.71       | 0.47     | 0.74     | 0.68  | 0.70   | 0.64        |
| I60         | Max. 2214   | 668.2      | 578.5    | 1054.7   | 1678 | 519.1  | 1163       |
|             | Min. 318.2  | 121.4      | 83.5     | 118.9    | 104.6 | 21.7   | 97.8        |
|             | Median 1209 | 229.8      | 409.5    | 533.1    | 939.8 | 279.6  | 690.3       |
|             | Mean 1213   | 231.5      | 412.1    | 537.2    | 940.1 | 278.9  | 692.7       |
|             | SD 774.6    | 242.5      | 207.3    | 383.2    | 643.0 | 203.1  | 436.3       |
|             | CV 0.64     | 1.05       | 0.50     | 0.71     | 0.68  | 0.73   | 0.63        |
| MSS         | Max. 1655   | 538.4      | 623.8    | 504.7    | 1668 | 515.2  | 1168       |
|             | Min. 248.9  | 98.6       | 98.7     | 73.6     | 99.7  | 20.4   | 99.1        |
|             | Median 884.2| 170.1      | 578.6    | 115.8    | 932.7 | 260.3  | 687.3       |
|             | Mean 886.3  | 171.3      | 582.7    | 117.4    | 933.6 | 261.4  | 689.9       |
|             | SD 575      | 118.8      | 249.0    | 202.2    | 640.9 | 202.0  | 438.5       |
|             | CV 0.65     | 1.16       | 0.43     | 1.72     | 0.69  | 0.77   | 0.62        |

TN, total nitrogen; Org-N, organic nitrogen; TP, total phosphorus; Org-P, organic phosphorus; SD, standard deviation; CV, coefficient of variance.

As observed in the chemical and physical analyses of TN and TP in Table 2, the data from each measurement from RSS to MSS also significantly varied. The forms of N and P during the digestion process can be determined by pH, rate and duration time of aeration, and temperature. Org-N and ammonium NH$_4^+$, the most abundant form of nitrogen in SS, can be converted into NH$_4^+$ by hydrolysis and NH$_3$, in the presence of moisture and alkaline pH condition, respectively. The formation of ammonia, a highly volatile substance, results in its release into the air. Petersen et al. [37] reported that the NH$_4^+$-N is nitrified to NO$_3^-$ in the presence of O$_2$. Nitrification can occur with dissolved oxygen concentration of above 1–2% of saturation and with a residence time of more than three days while denitrification can occur when the dissolved oxygen concentration is less than 10–15% of saturation [38,39]. Denitrification is a process in which nitrogen oxides (NO$_3^-$ and NO$_2^-$) are reduced stepwise to gaseous forms (NO, N$_2$O, and N$_2$). Under anaerobic conditions, nitrogen oxides are used as alternative electron acceptors to oxygen, resulting in substantial loss of N emitted as N$_2$O and N$_2$ in anoxic conditions [40,41]. As a consequence, there is simultaneous nitrification and denitrification possible depending on the dissolved oxygen concentration between 1 and 10% of saturation. Moreover metals and acidic condition in SS are detrimental inhibition factors to determining the form and its content of N during the digestion process [13,20,25,30,32].
The mean contents of TN, Org-N, NH$_4^+$-N, and NO$_3^-$-N in RSS were 2908, 1285, 49.8, and 1641 mg L$^{-1}$, respectively. However, those of TN and Org-N in FSS were sharply decreased to 2149 and 533 mg L$^{-1}$, respectively, while those of NO$_3^-$-N and NH$_4^+$-N were similar to those of RSS. The reduced proportion of TN and Org-N between RSS and FSS corresponded to 26.1% and 58.4% of the contents of RSS and the reduced proportion of Org-N corresponded to almost 99% of TN in RSS. This proved that the decrease in TN was attributed mainly to a decrease in Org-N which was present and separated from the RSS as a solid fraction in the SS. During the aeration processes of the three cells from FSS to MSS, the mean contents of TN, Org-N, and NH$_4^+$-N were decreased from 2149 to 886.3 mg L$^{-1}$, 533.4 to 171.3 mg L$^{-1}$, and 1578 to 117.4 mg L$^{-1}$, respectively, while the mean contents of NO$_3^-$-N was rapidly increased from 49.8 to 582.7 mg L$^{-1}$. The reduced proportion of mean Org-N and NH$_4^+$-N were approximately 28.2% and 67.9% and 89.1% and 92.6% for mean Org-N and NH$_4^+$-N measured in RSS and FSS, respectively. However, the sum of Org-N and NH$_4^+$-N reduced between FSS and MSS was greater than the mean content of TN decreased between FSS and MSS. The difference of reduction between TN and the sum of Org-N and NH$_4^+$-N could be compensated by an increase in NO$_3^-$ content. However, the reduction in TN between FSS and MSS should be interpreted mainly by N loss as NH$_3$ which could be volatilized in an alkaline pH range over three aerated digestion cells from FSS to MSS. Indeed, Bicudo and Svoboda [30] ascribed the increase in the concentration of NO$_3^-$ during aerobic digestion of SS to the accelerated conversion of NH$_4^+$-N to NO$_3^-$ because of the elevated activities of nitrifying bacteria. Additionally, Bicudo and Svoboda [30] indicated that a significant portion of N (approximately 10.5%) was denitrified by the end of intermittent aerobic digestion, which may also account for the observed losses in both TN and NH$_4^+$-N. In a comparison of N fractions in FSS and MSS, the dominant N fraction was NH$_4^+$ by 73.4% in FSS while NO$_3^-$ was dominant in MSS by 65.7%. The observed loss in TN and NH$_4^+$-N was a result of gaseous emissions under alkaline pH and aeration over three digestion processes, especially NH$_3$ and N$_2$O, as reported by Calvet et al. [42] and Mostafa et al. [43,44], but also by the mineralization of the latter to NO$_3^-$. The studies on aeration that consider the fate of N in SS revealed that continuous or intermittent aeration achieves high nitrogen removal as emissions of NH$_3$, N$_2$O, and N$_2$ [42]. The amount of N$_2$O emissions, which mainly occur in SS with natural crusts, is very small [45]. IPCC [46] estimated that the average N$_2$O-N loss from slurry storage with natural crusts is 0.5% of TN stored, with an uncertainty range of 0.25–1% of TN, and there is no N$_2$O-N loss from slurry storage without natural crusts. Therefore, we assumed that the amount of N$_2$O emission was very little during the digestion process between FSS and MSS.

As discussed above, the forms of P in SS are Org-P and inorganic P as solution phase that can be influenced by the decomposition of Org-P depending on pH and aeration during the digestion process. Li et al. [47] observed that a substantial increase in orthophosphate in the first day of aeration was observed in both the intermittent and continuous aeration treatments because both the organic and soluble P were decreased in the first day. The increase of insoluble inorganic P results from the chemical precipitation of the raised pH [7,10,21,24,34,41,44,45]. Thus, the forms and their contents of P in SS may vary with conditions such as pH, redox potential, temperature, and so on. The changes of mean contents of TP and Org-P between FSS and MSS were from 984.2 to 933.6 mg L$^{-1}$, and 312.8 to 261.4 mg L$^{-1}$, respectively, while that of PO$_4^{3-}$-P was from 668.4 to 689.9 mg L$^{-1}$. Although there was an increase in the PO$_4^{3-}$-P, this could not compensate for the net lost P. Phosphorus and multivalent metal ions cations such as Ca$^{2+}$ and Mg$^{2+}$ in solution can produce metal precipitates in addition to organically bound phosphorus and some residual polyphosphate [48]. For example, the precipitation of calcium phosphate from slightly alkaline wastewater can be approximately represented as the precipitation of hydroxyapatite [SCa$^{2+}$ + 3PO$_4^{3-}$ + OH$^-$ = Ca$_5$(PO$_4$)$_3$OH(s)]. Therefore, the formation of precipitate can depend on the amount and types of multivalent metal ions, the content of organic matter, including dissolved organic components, and solution pH. The reduction in TP between
RSS and FSS was caused by the separation process of solid particles including Org-P and chemical precipitates of P by flocculation using chemicals [47], while the reduction in Org-P from FSS to MSS could be caused by transformation into inorganic P [49,50]. However, a net loss of TP can be explained by precipitation of inorganic P from FSS to MSS, although the amount of precipitate can be governed by solution pH and the amount and types of multivalent metal ions in the solution phase at each corresponding aerated digestion stage.

Figures 4 and 5 show the changes in the mean proportions of the different types of N and P corresponding to each digestion stage from RSS to MSS. The proportions of TN, Org-N, and NH₄⁺-N were decreased with increasing pH, respectively, while the proportion of NO₃⁻-N was steadily increased with increasing pH from RSS to MSS. However, a significant content of Org-N reduced between RSS and FSS did not appear cause an increase in other forms of N in FSS: this can be interpreted as net loss from SS by separation of solid substances including OM. The decreasing of contents and forms of Org-N and NH₄⁺-N from FSS were relatively more rapid than those of NO₃⁻-N over three digestion cells. The proportions of Org-N, NH₄⁺-N, and NO₃⁻-N in MSS as a final product were 19.3%, 13.2%, and 65.7%, respectively, representing that NO₃⁻-N is the dominant form of N contrary to the proportion distributions of RSS and FSS. With these results, we could conclude that the content and forms of N in SS could be strongly influenced by solution pH of SS and aeration rate during the digestion process.

The changes of contents and forms of P depending on the digestion processes are presented in Figure 5. The changes in proportion of TP and Org-P were relatively rapid between RSS and FSS, and then slightly increased between FSS and MSS, while the proportion of PO₄³⁻-P was slightly higher or similar to that of MSS. The proportion of Org-P was decreased with increasing pH, while the proportion of PO₄³⁻-P was similar over the digestion processes. The net difference of P between TP and the sum of Org-P and PO₄³⁻-P could be explained by precipitation of P with metal species in SS depending on pH and aeration. PO₄³⁻-P converted from Org-P can be incorporated into SS, which can compensate the loss of P precipitated with metal species in a given pH. Therefore, the proportion of PO₄³⁻-P as the dominant P species can be recovered in MSS.

![Figure 4](image-url)

**Figure 4.** Changes in proportion of N content and pH corresponding to each digestion stage. TN, total nitrogen; Org-N, organic nitrogen; NO₃⁻-N, nitrate; NH₄⁺-N, ammonium. Error bars indicate standard error.
The results of Pearson correlation tests amongst pH, TN, NO$_3^-$-N, NH$_4^+$-N, OM, aeration for N and pH, TP, Org-P, PO$_4^{3-}$-P, and aeration for P are presented in Table 6. These correlations allowed us to identify tendencies in the content and type of N and P during the digestion of SS. Among the positive correlations, a strong interdependence occurred between Org-N and OM (0.999) for N and TP and Org-P (0.999) for P, respectively.

**Table 6.** Pearson’s correlation coefficients across types of N and P, pH, and OM in slurry characteristics.

| Measure          | Nitrogen (N) | Phosphorus (P) |
|------------------|--------------|----------------|
|                  | pH           | TN             | NO$_3^-$-N | NH$_4^+$-N | OM         | Aeration | pH           | pH           | TP           | Org-P       | PO$_4^{3-}$-P | OM         | Aeration |
| pH               | -0.954       | 0.944          | -0.923     | 0.962      | -0.795     | 0.443     | pH           | -0.648       | 0.673       | 0.915       | -0.795     | 0.443     |          |
| TN               |              |                |           |           |            |           |              |              |             |             |            |           |          |
| NO$_3^-$-N       | -0.745       | -0.779         | 0.909     | -0.305     | -0.523     | -0.773    | 0.959        | -0.748       | 0.676       | 0.876       | -0.511     | -0.838    | 0.759     |
| NH$_4^+$-N       | -0.960       | -0.729         | 0.305     | -0.523     | -0.773     | 0.959     | -0.748       | 0.676       | 0.876       | 0.511       | -0.838    | 0.759     |
| OM               | 0.763        | -0.346         | 0.526     | 0.346      | 0.346      | 0.598     | 0.346        | 0.526        | 0.346       | 0.598       | 0.346     | 0.598     |

TN, total nitrogen; Org-N, organic nitrogen; NO$_3^-$-N, nitrate; NH$_4^+$-N, ammonium; OM, organic matter content; TP, total phosphorus; Org-P, organic phosphorus; PO$_4^{3-}$-P, phosphate.

For N, the higher correlation was followed by pH and NO$_3^-$-N (0.986), TN and Org-N (0.944), and TN and NH$_4^+$-N (0.942), while the highest negative correlations were observed in NO$_3^-$-N and NH$_4^+$-N (−0.998), followed by pH and NH$_4^+$-N (−0.992), and pH and TN (−0.954). From these correlations, we could assume that the presences of Org-N, NO$_3^-$-N, and TN were strongly influenced by OM, pH, and Org-N, respectively, while those of NO$_3^-$-N, NH$_4^+$-N, and TN showed less interdependence with pH, respectively. For aeration, all factors except pH and NO$_3^-$-N showed negative correlation. As we observed above, the content of NO$_3^-$-N was increased with increasing pH over a 3 day digestion period as well as the increasing content of Org-N with OM, while NO$_3^-$-N content was increased with decreasing NH$_4^+$-N in the SS with the correlations of 0.335 and 0.443 between aeration and NO$_3^-$-N and pH and aeration. This indicated that the influence of pH was stronger than aeration on the formation of NO$_3^-$-N.

For P, the higher correlation was followed by Org-P and OM (0.982) and PO$_4^{3-}$-P, and the highest negative correlation was observed from OM and PO$_4^{3-}$-P (−0.839), followed by PO$_4^{3-}$-P and Org-P (−0.773), and PO$_4^{3-}$-P and TP (−0.747). As observed in the correlation among the factors for N, aeration showed negative correlation with the factors except pH and PO$_4^{3-}$-P, while it showed relatively higher interdependence with pH and PO$_4^{3-}$-P during the digestion process. As shown in Figure 5, there was a sharp decrease in TP and Org-P between RSS and FSS without aeration, and then a slight increase in PO$_4^{3-}$-P.
during the aeration process from FSS to MSS. This indicated that the aeration factor did not significantly contribute to the increase in PO$_4^{3-}$-P over the three-cell aeration process.

4. Conclusions

There is a drastic reduction in TN and TP by the separation of solid particles from RSS and a significant reduction in N caused by emission of NH$_3$, N$_2$O, and N$_2$ during intermittently aerated digestion processes between FSS and MSS. This widened the N to P ratio; therefore, if the application rate of CLSS is based on the N content, it could lead to an overapplication of P. It was also evident from the study that, as the SS gets aerated as it goes through the different aeration cells for the 90 days, the gap between the nitrogen to phosphorus ratio widens. Therefore, it will be prudent to shorten the length of the aeration process to reduce the gap in the nitrogen to phosphorus ratio in the SS. The high EC of MSS is indicative of high concentrations of mineral elements, which also indicates a potential risk of secondary soil salinization and accumulation of heavy metals such as copper and zinc in soil with successive applications of CLSS. In addition to these problems, the most dominant form of available N in MSS is nitrate, which can be easily leached out of soils or transformed into the potent greenhouse gas as N$_2$O or N$_2$. The results indicate that the nutritional value and efficiency of MSS should be improved by reducing N loss during the solid particle separation and intermittently aerated digestion processes and by lowering P:N ratios of the final product to conserve valuable nitrogen and to increase the applicability of CLSS as a fertilizer.

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