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Research article

# Pig manure treatment and purification by filtration

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This study aimed to develop a new, complex pig manure treatment and filtration process. The final scheme, called the AMAK process, comprised the following successive steps: mineralization with mineral acids, alkalization with lime milk, superphosphate addition, a second alkalization, thermal treatment, and pressure filtration. The proposed method produced a filtrate with 95%, 80%, and 96% reductions in chemical oxygen demand, nitrogen content, and phosphorus content, respectively. An advantage of the proposed method was that it incorporated a crystalline phase into the solid organic part of the manure, which enabled high filtration rates (>1000 kg $m^{-2}$ $h^{-1}$) and efficient separation. The process also eliminated odor emissions from the filtrate and sediment. The treated filtrate could be used to irrigate crops or it could be further treated in conventional biological wastewater treatment plants. The sediment could be used for producing mineral-organic fertilizer. The AMAK process is inexpensive, and it requires low investment costs.



## 1. Introduction

The pig manure produced by intensive pig farming has negative effects on the environment. The nitrogen and phosphorus compounds in the manure contaminate the soil and bodies of water. Stored manure generates odor, primarily due to the anaerobic decomposition of proteins (De la Torre et al., 2000). The chemical composition of pig manure depends on many factors, including the type and age of the animals and the feeding method (Sánchez and González, 2005). Manure is a two-fraction mixture of urine, feces, and water. The liquid fraction principally contains nitrogenous compounds (including ammonia, ammonium compounds, nitrates), and organic matter (Bertora et al., 2008). The solid fraction principally contains phosphoric compounds, which mainly occur in inorganic form (74–87% of the total P content) and organic compounds (Lens et al., 2004). The nitrogen content of pig manure determines its value for fertilization (Fangueiro et al., 2012, Rulkens et al., 1998). However, the farmland area that uses pig manure for fertilization is decreasing in size, and there are problems with managing this waste (Basset and van der Werf, 2005).

Manure must be separated into solid and liquid fractions before it can be treated and utilized. Many pig slurry separation techniques influence the characteristics of N in the resulting liquid and solid fractions, which may alter its potential availability to plants (Fangueiro et al., 2012). The simplest technology, physical separation, can remove up to 80% of the total solids from livestock manures (Hjorth et al., 2010, 2008; Melse and Verdoes, 2005). Current separation processes have some limitations. The biggest problem is the rather high investment and processing cost (Burton, 2007). By treating pig manure with flocculants before separation, the filtration efficiency was significantly improved (Pérez-Sangrador et al., 2012). Walker and Kelley (2003) evaluated the efficiency of pig slurry separation by gravity settlement before and after the addition of polyacrylamide (PAM) flocculants followed by screening. These resulted in removing constituents from the liquid fraction with following removal rates: 73% of total solids (TS), 87% of volatile solids, 71% of chemical oxygen demand (COD), 40% of total Kjeldahl nitrogen (TKN), and 34% of soluble phosphorus. Chelme-Ayala et al. (2011) treated pig manure with a physical-chemical treatment that included coagulation, flocculation, and sedimentation, followed by an oxidation step as a polishing treatment performed at the bench-scale level. That process resulted in an 82% reduction in the initial $NH_3$ and a 78% reduction in the initial total organic carbon.

The BIOSORTM-Manure biofiltration process (Buelna et al., 2008) for treating pig manure maintained the following overall pollutant removal rates: >95% of $BOD_5$, >97% of suspended solids (SS), >84% of TKN, and >87% of P, despite strong variability in $BOD_5$ (10–20 g $L^{-1}$), SS (10–20 g $L^{-1}$), TKN (2.0–3.8 g $L^{-1}$), and P (0.5–0.9 g $L^{-1}$). This process also eliminated >80% of the odor intensity from production units and from manure storage.

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The BIOREK process (Du Preez et al., 2005), which has been tested on a pilot scale, included anaerobic digestion, ammonia stripping, ultrafiltration, and reverse osmosis, and its operational costs were high. This process could achieve ammonia removal efficiencies of up to 99.9%.

The SELCO-Ecopurin (Martinez-Almela and Barrera, 2005) separation technology has been used for five years on 12 livestock farms in Spain, Italy, and the USA. The high recovery of solids (>90%) made it economical to use advanced purification techniques for the liquid. Karakashev et al. (2008) tested different processes for reducing organic matter in pig manure. In their final scheme (PIGMAN concept), the following successive process steps were implemented: thermophilic anaerobic digestion with separation by decanter centrifugation; post-digestion in an up-flow anaerobic sludge blanket UASB reactor; oxygen-limited autotrophic nitrification–denitrification process OLAND. This combination of steps reduced the filtrate contents of total organic matter by 96%, nitrogen by 88%, and phosphorus by 81%. This filtrate could be submitted to further cleaning treatments, or could also be directly used to irrigate crops.

After pig manure separation, the solid fraction can be used as an organic fertilizer or to generate power in biomass incineration plants and agricultural biogas generators (Hjorth et al., 2010; Ndegwa, 2001). The liquid products of the separation process can be treated further with evaporation, membrane filtration, or ammonia stripping; however, to date, no low-maintenance and/or cost-efficient applications of these post-treatments have been demonstrated (Møller et al., 2000). Melse and Verdoes (2005) described microfiltration of the centrifuged filtrate. Fugere et al. (2005) described nanofiltration to treat the overflow from settling processes. With these approaches, 33% of the initial crude slurry volume was obtained, and the final permeate was suitable for reuse as sanitary, safe industrial water. Ledda et al. (2013) studied nitrogen and water recovery from animal slurries with a process that integrated ultrafiltration, reverse osmosis, and cold stripping. Pieters et al. (1999) also implemented reverse osmosis at the farm scale.

Jørgensen and Jensen (2009) investigated the chemical and biochemical properties of solids collected from 47 commercial pig manure slurry separation plants that separated liquid manure into a nutrient-rich solid fraction and a nutrient-poor liquid fraction. The samples originated from five different types of separation technologies that processed pig manure and anaerobically digested manure. The largest variations found in the measured chemical and biochemical characteristics of the samples were the contents of ash, total P, total C, the SS, and the C distribution in the biochemical fractions.

The present study aimed to develop a new pig manure treatment and filtration process. Previously, a method was proposed that attained a removal efficiency of approximately 90% for COD, >99% for TS, and up to 47% for TKN (Kowalski et al., 2013c, 2012, 2014). The mineralization process eliminated ~75% of the odor intensity of the pig manure. However, a disadvantage of this elaborate method was that the P content was increased in the filtrate, reaching 5–6-fold of the content in raw pig manure (Kowalski et al., 2013c). The goal of the present study was to develop new complex mineralization and filtration process allowed eliminating the phosphorus content in the filtrate and improves the COD and N removal efficiency, and decreasing odor emission by selecting the most advantageous parameters in the treatment process.

## 2. Materials and methods

### *2.1. Characteristics of pig manure*

For this study, we used manure from a pig farm located near Piła, Poland. Previous studies (Kowalski et al., 2013a, 2013b, 2014) analyzed pig manure collected between June 2011 and May 2012 from this farm. The farm produced piglets intended for fattening at other pig farms, and sows were maintained for renewing the stock. The average monthly livestock statistics were as follows: 1101 sows, 64 gilts, 2536 sucking piglets, 140 weaned piglets, 200 shoats, and 160 porkers. The total number of pigs was 4201. Pig manure samples were collected from a drainpipe that carried slurry from the pig farm to a lagoon. For the treatment process, two representative 10-L samples of pig manure were marked “raw manure A” (sampled in May 2012) and “raw manure B” (sampled in June 2012). A total of 150–230 g of manure was processed in one batch. The “raw pig manure A” contained 11.5% TS and 8.65 g $L^{-1}$ TKN, 117.25 g $L^{-1}$ COD, and 2.42 g $L^{-1}$ P. The “raw pig manure B” contained 9% TS and 5.25 g $L^{-1}$ TKN, 92.25 g $L^{-1}$ COD, and 1.36 g $L^{-1}$ P.

### *2.2. Methods for removing organic matter, nitrogen, phosphorus, and odor*

After stirring, the samples were diluted with water to achieve a TS content of 2%–10% (Table 1) (Kowalski et al., 2014). Next, we added 75% technical grade phosphoric acid (Brenntag, Kędzierzyn, Poland) and 95% technical grade sulfuric acid (KGHM Metraco S. A., Legnica, Poland) to the manure samples to obtain pH values of 5.5 and 3.0, respectively. Next, the slurry was alkalized with a 10% solution of lime milk (calcium hydroxide solution contained 10% of CaO, produced from technical grade, 95% CaO; Lhoist SA., Poland), to obtain a pH from 9.0 to 10.5; then, we added superphosphate (Luvena SA., Luboń, Poland) in amounts that comprised 3%–8% of the initial manure weight (Table 2) and the mixture was again alkalized with lime milk to obtain a pH from 9.0 to 10.5 (Table 3). The processed slurry was heated for ~50 min at ~90 °C, and filtered with a pressure filter under pressures up to 0.3 MPa, at 70–75 °C. For the laboratory tests, we used a Sartorius AG pressure filter (Goettingen, Germany), with a volumetric capacity of 2 L. In the filtrate we determined the filtrate pH, COD load, N and P content. We also determined the sediment moisture content and chemical composition, and we conducted elementary analyses of sediment P, Ca, Mg, S, K, N, C, and H contents (Kowalski et al., 2013a, 2013c, 2014).

### *2.3. Analyses*

For the Kjeldahl method of nitrogen determination, we used a DK6 mineralizer. We determined the phosphorus content with a nanocolor UV/VIS spectrophotometer. Samples were mineralized for COD determination with an M-9 mineralizer. To determine the Ca, K, Mg, P, and S contents in the sediment, we used an inductively-coupled plasma atomic emission (ICP-AE) spectrometer OPTIMA 7300 DV (Perkin Elmer, Waltham, MA, USA). The C, H, and N contents were determined with a PE 2400 analyzer (Perkin Elmer, Waltham, MA, USA). We determined the phase composition of the sediments with a Bruker AXS D8 Advance diffractometer (BRUKER AXS, Inc., Madison, WI, USA). With the Rietveld method and the semi-quantitative analysis software package, RayfleX Autoquan, version 2.6 (Taut et al., 1998) we determined the contents of the crystalline and amorphous phases We performed microscopic analyses of the sediments with a Hitachi TM 3000 electron microscope. The chemical composition of the pig manure was determined in accordance with Polish standards for the examination of waste and fertilizers (Polish Standard, 2006, 2001a, 2001b, 1993). The microbiological analyses were conducted in accordance with the Polish standard (2003). The odor intensities were measured in the gases sampled from the pig manure before treatment and from the filtrates and sediments obtained after treatment