

# Preliminary Study on Processing Pig Manure with the Filtration Method and the Possibility of Applying the Post-Filtration Sediment as a Potential Fertilising Agent

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#### Summary

Problems with storage and management of manure from pig farming on the one hand and possibility to use the manure as a renewable source of fertilizer components on the other hand encourage seeking new effective methods of manure management. For reduction of odor emission and costs of storage and transportation as well as for proper preparation of the manure for further treatment, the material has to be separated into solid and liquid fractions. Pig manure taken from one livestock farm in Poland was used in the experiment. The tests comprised mineralization of pig manure components with use of phosphoric and sulfuric acids, lime milk and super phosphate, heat treatment and subsequent filtration with use of pressure filter. Changing in the mineralization parameters of manure allowed obtaining after filtration sediment and filtrating with much diversified chemical and physical properties. In the after filtrate sediment N, P, K, Mg, Ca, K, C, H and dry mass content were analyzed. Phase composition of sediments was determined too. N content in the after filtrate sediment ranged from 0.79 to 1.56% and P content from 8.5 to 14.95%. Efficiency of the suspension filtration process varied within the range of 518–2627 kg/m<sup>2</sup>/h.

Keywords: pig manure treatment, process parameter, filtration process, liquid fraction, solid fraction

### Introduction

Manure is a natural liquid animal fertilizer constituting a mix of faces, urine, fodder remains and water used for elimination of faces from stock facilities (Chelme-Ayala et al., 2011; Girard et al., 2009; Møller et al., 2000). It is generated in the conditions of dry swine rising. Composition of the manure ranges broadly and depends on many factors, in particular on the type and age of animals, their feeding method and care, as well as volume of water used for sanitary and cleaning purposes, as well as storage method and time, and manure dilution (Girard et al., 2009; Gorlach and Mazur, 2001; Pawełczyk and Muraviev, 2003; Zhang and Westerman, 1997). Manure usually contains 8-10% dry mass and constitutes a source of valuable fertilizing agents: nitrogen, phosphorus, potassium and other (Pawełczyk and Muraviev, 2003). Average content of the main fertilizing agents in the manure is as follows: nitrogen 0.6%, phosphorus 0.2%, potassium 0.2% (Pawełczyk and Muraviev, 2003; Suresh et al., 2009).

Manure must be properly managed, as it can constitute a potential threat to the natural environ-

ment. The most frequent method of using the manure from a pig farm is its application as liquid organic fertilizer. The greatest impact on the fertilizing value of the manure is from animal feeding method, degree of faces dilution with water, and the degree of vegetable use of its nutrients. The principle must be observed not to exceed the annual dose of 170 kg total nitrogen per 1 hectare of farming land, which corresponds to 45 m<sup>3</sup> manure per hectare (Bertora et al., 2008; Buelna et al., 2008; Pawełczyk and Muraviev, 2003). It is not always possible to meet such conditions, in particular in the areas with small area of farming lands, and in the case of large industrial farms that do not feature manure containers with the appropriate capacity. In practice, this is related to significant problems with waste management, which can lead to excess pouring of the manure onto nearby fields (Buelna et al., 2008; Pawełczyk and Muraviev, 2003). The application of too high doses of manure many cause reduced capacity for seed sprouting and crop incubation, as well as prolongation of the vegetation period of plants and their reduced quality. From the ecological point of view, the greatest threat

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to the natural environment in the agricultural production is the washing out of nitrates to underground waters, which impacts on the quality of drinking water and surface flow of nitrates and phosphates, causing eutrophication of water containers (Buelna et al., 2008; Imbeach, 1998; Rulkens et al., 1998).

In order to reduce odor emission and the costs of manure storage and transport, as well as to improve its fertilization value and properly prepare it for further processing, it is justified to separate manure into solid and liquid fraction (González-Fernández et al., 2008; Hjorth et al., 2010; Møller et al., 2000; Zhang and Westerman, 1997). The selection of the appropriate technology for manure separation into fractions and the application of the relevant equipment are done on the basis of physical properties and chemical composition of the manure, as well as potential use of the products of separation (Girard et al., 2009; Hjorth et al., 2010). Among the methods for manure separation into solid and liquid fraction, there is the pressure filtration method, which allows for obtaining solid fraction with high dry mass content (Hjorth et al., 2010; Møller et al., 2000). Liquid fraction obtained as a result of separation can be directly applied to soil, subjected to oxygen processing, or further purified and applied for field sprinkling. Solid fraction can be used as organic fertilizer or soil improving agent, or subjected to composting and applied after relevant decomposition period, similarly as farmyard manure (Burton, 2007; Konieczny et al., 2011; Jorgensen and Jensen, 2009; Ndegwa et al., 2001). Of particular interest is the issue of using the solid fraction to obtain mineralorganic fertilizers used for agricultural purposes.

## Materials and methods

Pig manure taken from one livestock farm in Poland was used in the experiment. Pig manure samples were taken from drainpipe carrying slurry from the pig farm to a lagoon and then transported under cool conditions in tightly sealed plastic bottles to the laboratory where the analysis was conducted. Chemical composition of the pig manure was determined in accordance with the effective Polish standards using appropriate analytical, physical and chemical method (Kowalski et al., 2012). The samples were analyzed to determine the content of nitrogen, the biochemical oxygen demand, the chemical oxygen demand, and the content of phosphorus, potassium, calcium, and dry mass. To determine the Kjeldahl nitrogen, a VELP DK6 digester and a VELP steam distillation unit were used. Phosphorus was determined with a Macherey-Nagel Nanocolor UV/VIS spectrophotometer. A WSL M-9 digester was used to mineralize the samples to determine the chemical oxygen demand. Potassium and calcium were determined by flame atomic absorption spectroscopy (FAAS) using a Perkin Elmer OPTIMA 7300 DV apparatus. Microbiological tests were performed on the analyzed slurry to identify *Salmonella* bacilli and live parasite eggs (Kowalski et al., 2012). For laboratory tests, the pressure filter of volumetric capacity of 2000 mL manufactured by Sartorius was used for manure filtration.

For determination of Ca, K, Mg, P, S contents in filtered sludge, Inductively Coupled Plasma Atomic Emission (ICP-AE) spectrometer of OPTIMA 7300 DV type manufactured by Perkin Elmer was used; the contents of C, H and N were determined with use of Perkin Elmer's PE 2400 analyzer. Phase composition of the sludge was determined with use of Philips X'Pert diffractometer with PW W 1752/0 graphite monochromator.

Each time prior to the commencement of mineralization process, the manure was thoroughly stirred, and after stirring, the weighed amounts of phosphoric and sulfuric acids were added to the manure for obtaining pH value of approx. 5.04-5.52 and 2.7-4.5, respectively. After treatment with acids, the slurry was treated with 10% solution of lime milk for obtaining a pH value of approx. 7.74-10.05, then super phosphate in amount of 8-10% of the initial manure weight was added, and the mixture was second time neutralized with the use of lime milk. The resulting slurry was heated for approx. 25-55 minutes, cooled down to ~75°C, poured into pressure filter and filtered. As a result of filtration, light straw colored filtrate and sludge were obtained (Kowalski et al., 2012).

## **Results and discussion**

The results physical, chemical and microbiological analyses of pig manure are shown in Table 1. The parameters of pig manure treatment process and the consumption of raw materials in the experiment is shown in Table 2. The filtration process indices and the results of sludge analyses are given in Table 3 and 4, respectively. The phase composition of the sludge is shown in Table 5, and the photo of the sludge is provided in Figure 1.

In the process of manure mineralization, depending on the dry mass content and chemical composition (Table 1), the volume of phosphoric acid and sulfuric acid consumed as well as of lime milk changes. The studies indicate that the greater the dry mass content in the manure, the greater consumption of the acids and lime milk (Table 2). The change to the dry mass content is also decisive for the quantities of filtration products obtained, namely sediment and filtrate (Table 3). The greatest volume of sedi-

Tabeta T. Analiza właściwości fizycznych, chemicznych i mikrobiologicznych obornika								
Determined	Manure batch							
parameter	Ι	II	III	IV	V	VI	VII	
$TKN^{1}$ (mg/L)	4370	5390	3230	8730	3680	7972	3960	
$(BOD_5)^2 (mg/L)$	25250	14500	7050	36900	9620	41400	12240	
$\text{COD}^3$ (mg/L)	53400	29600	14950	87900	20000	98300	36880	
Phosphorus (mg/L)	910	554	221	1660	462	1810	784	
Potassium <sup>4</sup> (%)	4.75	7.3	3.77	1.95	3.37	6.92	6.8	
Calcium <sup>4</sup> (%)	3.27	3.43	5.2	2.24	5.28	2.3	3.36	
Dry matter (%)	4.05	3.3	2.9	10.95	2.1	9.48	1.7	
Salmonella group bacilli (amount/L)	not found	not found	not found	not found	not found	not found	not found	
Parasite eggs <sup>5</sup> (amount/L)	none	none	none	none	none	none	none	

Table 1. Physical, chemical and microbiological analysis of pig manure Tabela 1. Analiza właściwości fizycznych, chemicznych i mikrobiologicznych obornika

<sup>1</sup> Total nitrogen determined with Kjeldahl's method

<sup>2</sup> Biochemical Oxygen Demand determined according to the dilution method (BOD<sub>5</sub>)

<sup>3</sup> Chemical Oxygen Demand (COD)

<sup>4</sup> Dry weight

<sup>5</sup> Parasite eggs (Ascaris sp., Trichuris sp., Toxocara sp.)

#### Table 2. Indices of raw material consumption in the manure treatment process

Consumed amount	of	Manure batch						
materials		Ι	II	III	IV	V	VI	VII
Manure (g)		253.44	139.42	140.66	150.18	150.82	230.46	230.61
Wallare (g)		pH 7.78	pH 8.13	pH 8.82	pH 7.45	pH 8.07	pH 7.57	pH 7.71
H <sub>3</sub> PO <sub>4</sub>		7.30	6.39	4.49	4.09	4.24	6.49	5.93
(pure – 75%) (g)		pH 5.47	pH 5.04	pH 5.04	pH 5.51	pH 5.51	pH 5.52	pH 5.51
H <sub>2</sub> SO <sub>4</sub> (techn. 95%) (g)		2.18 pH 3.10	1.31 pH 2.7	0.49 pH 2.96	2.25 pH 4.50	0.73 pH 2.98	2.82 pH 3.01	1.91 pH 2.99
Lime milk	Ι	36.42 pH 8.41	32.08 pH 9.0	18.9 pH 8.81	38.63 pH 9.03	30.39 pH 10.05	42.46 pH 8.55	24.64 pH 8.49
(10% solution) (g)	Π	24.21 pH 7.74	8.94 pH 7.74	17.08 pH 8.41	20.52 pH 8.15	17.25 pH 8.02	10.81 pH 8.04	18.49 pH 8.04
Super phosphate (g)		25.34 pH 4.92	13.94 pH 5.80	14.06 pH 4.95	15.02 pH 6.64	15.08 pH 7.05	18.44 pH 5.78	18.45 pH 4.64
Heating time [min]		40	25	35	45	45	55	50

Tebela 2. Wskaźniki konsumpcji materiału surowego podczas procesu obróbki obornika

Table 3. Filtration process indices

Tabela 3. Wskaźniki procesu filtracji

Determined parameter	Manure batch							
Determined parameter	Ι	II	III	IV	V	VI	VII	
Actual weight of the manure after treatment delivered to filtration (g)	329.54	166.29	148.10	196.08	157.65	256.96	252.14	
Amount of the sludge obtained (g)	75.75	37.68	37.42	102.89	45.1	75.63	52.47	
Amount of the filtrate obtained (g)	238.78	123.18	105.82	85.39	105.65	177.71	195.24	
Filtration time (s)	38	16	25	18	27	111	24	
Filtration rate (kg/m <sup>2</sup> /h)	2060	2598	1322	2451	1314	518	2627	

ment, amounting to approx. 103 g, was obtained using manure containing 10.95% dry mass. Efficiency of the suspension filtration process varied within the range of  $518-2627 \text{ kg/m}^2/\text{h}$ . The lowest efficiency was obtained in the case of manure VI, where addition of phosphoric acid(V) and sulfuric acid(VI) was applied to pH of 5.52 and 3.01, respectively, and then the addition of lime milk at the first

Table 4. Results of filtered sludge analyses
Tabela 4. Wyniki analizy przefiltrowanego szlamu

Determined parameter	Filtered sludge							
	Ι	II	III	IV	V	VI	VII	
C (%)	15.61	6.52	4.63	18.54	5.84	13.05	10.80	
H (%)	2.68	1.46	1.16	3.02	1.41	2.1	2.15	
N (%)	1.15	1.05	0.68	1.56	0.79	1.25	0.90	
Ca (%)	25.33	28.56	32.59	19.59	30.48	19.58	22.40	
K (%)	0.39	0.39	0.29	0.58	0.29	0.29	0.21	
Mg (%)	0.78	0.38	0.38	0.53	0.33	0.49	0.51	
P (%)	13.10	14.11	14.95	8.68	13.60	8.5	10.67	
S (%)	2.01	1.10	1.07	2.31	1.61	1.53	1.58	
Moisture content (%)	50.96	45.15	52.46	65.31	59.12	48.46	43.0	

Table 5. Phase composition of the filtered sludge

Tabela 5. Skład fazowy przefiltrowanego szlamu

No.	Phase composition
Ι	CaSO <sub>4</sub> ·0.5H <sub>2</sub> O; Ca <sub>2,86</sub> Mg <sub>0,14</sub> (PO <sub>4</sub> ) <sub>2</sub> ; Ca <sub>5</sub> (PO <sub>4</sub> ) <sub>3</sub> (OH)
II	$Ca_5(PO_4)_3(OH); CaHPO_4$
III	$Ca_5(PO_4)_3(OH); CaHPO_4$
IV	$Ca_5(PO_4)_3(OH)$ ; $CaHPO_4 \cdot 2H_2O$ ; $SiO_2$
V	Ca <sub>5</sub> (PO <sub>4</sub> ) <sub>3</sub> (OH); CaHPO <sub>4</sub> ·2H <sub>2</sub> O; CaSO <sub>4</sub> ·2H <sub>2</sub> O; SiO <sub>2</sub>
VI	$Ca_{5}(PO_{4})_{3}(OH)$ ; $CaSO_{4} \cdot 2H_{2}O$ ; $CaHPO_{4} \cdot 2H_{2}O$ ; $CaHPO_{4}$ ; $Ca_{18}Mg_{2}H_{2}(PO_{4})_{14}$ ; $SiO_{2}$
VII	$Ca_5(PO_4)_3(OH)$ ; $CaSO_4 \cdot 2H_2O$ ; $CaHPO_4$ ; $Ca_{18}Mg_2H_2(PO_4)_{14}$ , $SiO_2$



Fig. 1. Photo of filtered sludge Rys. 1. Zdjęcie przefiltrowanego szlamu

stage to pH of 8.55, and at the second stage to pH 8.04. The highest filtration efficiency was obtained in the case of applying manure containing the lowest dry mass content (1.7%), while applying similar mineralization parameters as in the case of sample VI, where only the heating time was shortened.

Moisture of post-filtration sediments obtained varied within the range of 43–65%. The main crystalline component of all post-filtration sediment samples was hydroxyapatite. The highest content of N, S, K, H and C was recorded in sediment obtained from manure containing 10.95% dry mass. The content of P and Mg in the sediment varied within the range of 8.5–14.95%, 0.33–0.78%. It is difficult to draw clear conclusions as to the change of mineral composition of post-filtration sediments obtained, as manure with different chemical composition was used, and the parameters of manure mineralization were changed. An important feature of

the operations performed is the fact of obtaining an after filtration sediment with moisture content allowing for mixing it with fertilizing agents allowing for obtaining a farming fertilizer that would meet standard requirements. An important issue is also reduction of odor emission level from filtration products.

## Conclusions

Pressure filtration is an effective method for manure fractioning. The presented preliminary studies allow for separating manure into solid and liquid fraction with filtration efficiency of 518–2627 kg/m<sup>2</sup>/h. The degree of filtration efficiency can be controlled by appropriate selection of the volume of solid phase in manure designed for processing, as well as mineralization process parameters. The main crystalline phase of the post-filtration sediment is hydroxyapatite. The content of fertilizing agents N, P and K in the post-filtration sediment varied within the range of: 0.68–1.56%, 8.5–14.95% and 0.21–0.58%. Balancing of sediment composition with the appropriate fertilizing agents creates an opportunity of obtaining agricultural fertilizers.

#### Acknowledgements

This study has been carried out in the framework of the Development Project No.14-0003-10/2010 granted by the National Centre for Research and Development.

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## Wstępne badania nad przeróbką gnojowicy świńskiej z wykorzystaniem filtracji i możliwością zastosowania sedymentu jako nawozu

Problemy ze składowaniem i gospodarką obornika pochodzącego z hodowli świń z jednej strony i możliwość wykorzystania nawozu jako odnawialnego źródła składników nawozu z drugiej zachęca do poszukiwań nowych efektywnych metod zarządzania obornikiem. W celu redukcji emisji odoru i kosztów składowania i transportu jak również odpowiedniego przygotowania obornika do następnych obróbek materiał został podzielony na frakcje stałe i płynne. Obornik został pobrany z polskiego gospodarstwa rolnego zajmującego się hodowlą świń i został użyty w eksperymencie. Testy obejmowały mineralizację składników obornika przy użyciu kwasu fosforowego i siarkowego, mleka wapiennego oraz superfosfatu, obróbkę termiczną oraz późniejszą filtrację za pomocą filtra ciśnieniowego. Zmiany parametrów mineralizacji obornika pozwoliły otrzymać osad pofiltracyjny i filtrowanie z dużo bardziej zróźnicowanymi właściwościami chemicznymi i fizycznymi. W osadzie znajdowały się N, P, K, Mg, Ca, K, C, H oraz materia sucha, które zostały poddane analizie. Określono również skład fazowy osadów. Zawartość N w osadzie wynosił od 0,79 do 1,56% a zawartość P od 8,5 do 14,95%. Wydajność procesu filtracji wahała się w zakresie 518 – 2627 kg/m<sup>2</sup>/h.

Słowa kluczowe: przeróbka gnojowicy świńskiej, parametry procesu, proces filtracji, frakcja ciekła, frakcja stała