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Pig manure treatment by filtration*

Zygmunt Kowalski[1]✉, Agnieszka Makara[1], Dalibor Matýsek[2], Józef Hoffmann[3] and Krystyna Hoffmann[3]

[1]Cracow University of Technology, Kraków, Poland; [2]VŠB-Technical University of Ostrava, Ostrava-Poruba, Czech Republic; [3]Wrocław University of Technology, Wrocław, Poland

A study of new pig manure treatment and filtration process was carried out. The advantage of the worked out technology is the method of incorporation of crystalline phase into solid organic part of manure. The obtained new solid phase of manure contains about 50% of crystalline phase forming a filtration aid that enables high effectiveness of manure filtration. The filtration rate of manure separation into solid and liquid fractions with pressure filter may achieve 1300–3000 kg/m²/h. The method makes it possible to maintain an overall average pollutant removal performance 90% for the chemical oxygen demand COD, >99% for the suspended solids SS, to 47% for the total nitrogen content. The obtained results showed that the proposed technology being efficient and simple offers a possible solution to pig manure problems.

Key words: pig manure treatment, filtration process, COD decrease

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INTRODUCTION

The manure produced in the bedding-free pig farming is a blend of animal droppings, fodder residuals and water used for keeping the piggery clean and tidy. The composition of the manure depends on many factors, particularly on the species and age of animals, the manner of their feeding and keeping, the amount of water used and the duration of manure storage and dilution. Nitrogen, present in manure in various forms, is a component that determines the value of the manure for fertilization (Girard *et al*., 2009; Rulkens *et al*., 1998; Sánchez & González, 2005; Zhang & Westerman, 1997). The nitrogen and phosphorus compounds from the manure contaminate soil and water courses. Volatile organic and inorganic compounds comprised in the manure cause emission of odor into the air (Pawełczyk & Muraviev, 2003).

Pig manure produced in high-density livestock farming needs proper management methods. In order to reduce odor emission, costs of storage, transportation and to properly prepare it for further treatment, the manure has to be separated into solid and liquid fractions (Hjorth *et al*., 2008; Hjorth *et al*., 2010). Separation processes have a distinct role in the management of livestock slurries, but it is important to recognize their limitations (Burton, 2007). The complete removal of all suspended matter of an effluent is theoretically possible by settling but the clarified stream still retains a significant polluting potential in terms of the residual nitrogen content and potassium.

Treatment with flocculants before separation improves its efficiency significantly. The products of the solid–liquid separation may be further treated by evaporation, membrane filtration or ammonia stripping in order to obtain the desired end-products; however, low-maintenance and/or cost-efficient operation of these post-treatments not been demonstrated yet (Moller *et al*., 2000). A study of flocculants addition on different livestock wastewaters was carried out in the work of Gonzalez-Fernandez *et al*., 2008. A range of 80–200 ppm of polyacrylamide (PAM) followed by screening was employed in the case of flocculation treatment. The removal rates in the liquid fraction were 73% for total solids, 87% for volatile solids, 71% for chemical oxygen demand, 40% for total Kjeldahl nitrogen, and 34% for soluble phosphorus.

Chemical and biochemical properties were investigated in 47 solids collected from commercial solids separation plants separating liquid manure into a nutrient-rich solid fraction and a nutrient-poor liquid fraction (Joergensen & Jensen, 2009). The samples originated from five different types of separation technologies, separating swine manure and anaerobically digested manure. The largest variations in measured chemical and biochemical characteristics between samples were found for ash, total P, total C, SS and C distribution in the biochemical fractions. The principal component analysis of the obtained data showed that the chemical and biochemical characteristics of the solids were dependent on the type of technology used for separation. In the work of Walker & Kelley, 2003 swine slurry solids separation efficiency through gravity settling was evaluated before and after the addition of a proprietary polymeric (PAM) flocculant. The results indicated that at concentrations of 62.5–750 mg/l the polymer improved slurry solids separation efficiency and significantly reduced concentrations of other associated aquatic pollution indicators. The use of polymer might facilitate further treatment and/or disposal and therefore reduce associated environmental degradation potential.

Different processes for reduction of the organic matter (anaerobic digestion, effluent separation by decanter centrifugation, membrane microfiltration, post digestion in up flow anaerobic sludge blanket (UASB) reactor, partial oxidation), nitrogen (oxygen-limited autotrophic

✉e-mail: zkow@chemia.pk.edu.pl

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Abbreviations: COD, chemical oxygen demand; TKN, total nitrogen determined with Kjeldahl's method; LF, liquid fractions; SF, solid fractions; BOD_5, biochemical oxygen demand; SS, suspended solids; P_{tot}, content of total phosphorus.

nitrification–denitrification, OLAND) and phosphorus (phosphorus removal by precipitation as struvite, PRS) from pig manure were tested in the work of Karakashev & Schmidt, 2008. In a final scheme (PIGMAN concept) combination of the following successive process steps was used: thermophilic anaerobic digestion with sequential separation by decanter centrifuge, post-digestion in UASB reactor, partial oxidation and finally OLAND process. This combination resulted in reduction of the total organic, nitrogen and phosphorus contents by 96%, 88%, and 81%, respectively.

Subject to the quality parameters, the liquid filtration product may be submitted to further cleaning treatment or directly used for crop irrigation. Solid fraction may be used as organic fertilizer or for power generation in biomass incineration plants and agricultural biogas generators (Ferrer *et al*., 2009; Hjorth *et al*., 2010; Ndegwa, 2001). Liquid/solid separation process was based on the experiments carried out with the use of a commercial separator system applying a cationic polymer and a filter press separator (Miller *et al*., 2007). Anaerobic digestion process (AD) runs under thermophilic conditions with post-digestion under mesophilic conditions to extract the additional methane. Thermal pre-treatment process (TPT), applied to separated solid manure fraction prior to AD process, runs at 127°C to improve biogas yield. In the Danish case, water from the separated solid fraction of pig manure evaporates in the drying process until achieving 95% of dry matter content. The system can recover up to 72.5% of the heat used for evaporation in heat exchangers (Prapaspongsa, 2010).

The integrated system of centrifugation/two-step ultrafiltration/nanofiltration was used to recover water from pig slurry (Konieczny *et al*., 2011). PVDF UF (100 and 50 kDa), PES UF (10 and 5 kDa) and composite hydrophilic NF membranes (0.2 kDa) were used. The study showed that among all the discussed system configurations centrifugation–UF50–UF5–NF0.2 was the most effective. 33% of initial crude slurry volume was obtained as the final permeate. The finally obtained product was suitable to be reused as sanitary safe industrial water.

Research work to optimize the BIOSORTM-Manure, a biofiltration process for pig manure treatment, have been realized on the site of a piggery (Île d'Orléans, Québec, Canada) using a 400 m^3 biofiltration system (Buelna *et al*., 2008). Despite strong variations in BOD_5 (10 000–20 000 mg/L), in SS (10 000–20 000 mg/L), in TKN (2000–3800 mg/L) and in P_{tot} (500–900 mg/L), the BIOSORTM made it possible to maintain an overall pollutant removal >95% for the BOD_5 >97% for the SS >84% for the TKN and >87% for the P_{tot}. The process eliminates over 80% the odor intensity coming from the production units, the storage, the transportation and the spreading of the manure.

In the study (Chelme-Ayala *et al*., 2011) pig manure was treated by physic-chemical treatment, including coagulation, flocculation, and sedimentation followed by an oxidation step as a polishing treatment at a bench-scale level. A superabsorbent polymer (SAP) and a mineral and salt formulation able to generate molecular iodine were used as coagulant and oxidant agents, respectively. Following the SAP application, 82% of initial NH_3, 78% of initial total organic carbon, and 93% of the total coliforms were reduced using 40 mg/L of free iodine.

This work presents new pig manure treatment and filtration process. The tests comprised mineralization of pig manure components with the use of phosphoric and sulfuric acids and super phosphate, heat treatment and subsequent filtration with pressure filter. The use of mineral acids allowed to eliminate pathogens. The advantage of the worked out technology is the method of incorporation of crystalline phase into solid organic phase of the manure of crystalline phase. This resulted in high filtration rate with applied pressure filters.

MATERIALS AND METHODS

For laboratory tests the pressure filter of volumetric capacity of 2000 mL manufactured by Sartorius was used. For Kjeldahl's method of nitrogen determination, DK6 mineralizer and equipment for distillation with steam, both manufactured by VELP, were applied. Phosphorus content was determined with Nano color UV/VIS spectrophotometer manufactured by Macherey-Nagel. Mineralization of samples for COD determination was made with M-9 mineralizer manufactured by WSL. For determination of Ca, K, Mg, P, S contents in the sediment, 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 Perkin Elmer's PE 2400 analyzer.

Phase composition of the sediments was determined with Bruker AXS D8 Advance diffractometer. In the samples with semi-quantitative analytical method the content of crystalline and amorphous phases were determined using Rietveld method and semi-quantitative analysis software package RayfleX Autoquan version 2.6 (Taut *et al*., 1998).

Microscopic analysis of the after filtration sediment was carried out with Hitachi TM 3000 electron microscope. Thermal analysis of the dried sediments was made with the TG/DTA 7300 EXSTAR SII derivatograph. The measurements were performed in an air atmosphere within temperature ranges of 25–1000°C.

Eight batches of the pig manure taken from the same pig farm were used. For treatment process 150–230 g of manure was processed in one batch. Chemical composition of the pig manure was determined in accordance with the Polish standards using appropriate analytical, physical and chemical methods (Kowalski *et al*., 2012). Microbiological analyses were conducted in accordance with (Polish standard, 2003). The results are given in Table 1.

First stage of experiment comprised performance of preliminary tests aiming at selection of parameters of pig manure mineralization and phase separation. These tests showed the possibility of COD load reduction in the liquid phase by minimum 80% at filtration rate of minimum 0.5 $m^3/h/m^2$ of filtration surface area (Kowalski *et al*., 2012).

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 approximately 5.5 and 3.0, respectively. Next the slurry was treated with 10% solution of lime milk for obtaining a pH about 8.5, then super phosphate in amount of 10% of the initial manure weight was added, and the mixture was second time neutralized with lime milk. The processed slurry was heated for about 50 minutes, cooled down to about 75°C and filtered with a pressure filter. As a result of filtration, light straw colored filtrate and sediment were obtained. The analyses comprised determination of pH value, COD load, contents of N according to Kjeldahl, and P_{tot} in the fil-

Table 1. Physical, chemical and microbiological analyses of pig manure.

Determined Parameter	Manure batch							
	I	II	III	IV	V	VI	VII	VIII
TKN (mg/L)	3680	4370	5390	8730	3960	7972	2956	3230
BOD_5 (mg/L)	9620	25250	14500	36900	12240	41400	2620	7050
COD (mg/L)	20000	53400	29600	87900	36880	98300	8760	14950
Phosphorus P_{tot} (%)	462	910	554	1660	784	1810	224	221
Potassium (%)	3.37	4.75	7.3	1.95	6.8	6.92	11.8	3.77
Calcium (%)	5.28	3.27	3.43	2.24	3.36	2.3	2.32	5.2
Dry matter (%)	2.1	4.05	3.3	10.9	1.7	9.48	1.0	2.9
Salmonella group *bacilli* (amount/L)	not found	not found	not found	not found	not found	not found	not found	not found
Parasite eggs[a] (amount/L)	none	none	none	none	none	none	none	none

[a]Parasite eggs (Ascaris sp., Trichuris sp., Toxocara sp.).

Table 2. The pig manure treatment process figures and parameters.

Raw materials used (kg/1000kg of manure)	Manure batch							
	I	II	III	IV	V	VI	VII	VIII
H_3PO_4 (pure – 75%)	29.7	28.8	45.2	30.0	25.0	27.8	29.2	32.0
H_2SO_4 (technical 95%)	5.6	8.6	10.0	25.2	7.5	13.3	9.2	3.5
Lime milk (10% solution)	229.2	238.9	293.3	346.1	180.3	227.5	198.6	255.8
Superphosphate	100.0	100.0	97.4	100.0	100.0	100.00	100.0	100.0
Treatment products (kg/1000kg of manure)								
Manure after treatment before filtration	2190.8	1300.3	1086.6	1261.4	1089.3	1209.5	1114.3	1052.9
Sediment	503.6	298.9	390.1	560.6	224.6	350.0	318.4	266.0
Filtrate	1587.4	942.0	646.6	669.5	830.0	834.6	755.2	752.3
Filtration rate (kg/m^2/h)	3043	2060	862	698	2015	1024	1461	1322

trate. Moisture content and chemical composition of the sediment were determined. Elementary analyses of P, Ca, Mg, S, K, N, C and H contents in the sediment were carried out, too.

RESULTS AND DISCUSSION

The treatment with mineral acids was aimed at transformation of macro-and micro- fertilizer components into the form bio-available to the plants, binding volatile organic and inorganic nitrogen compounds and hydrolysis of organic matter. Moreover, the addition of acids improves sanitary and epidemic safety owing to elimination of pathogens (bacteria and parasite eggs). The manure slurry was neutralized with lime milk (10% solution) at two stages of the treatment. The addition of super phosphate-type fertilizer allowed also balancing nitrogen and phosphorus contents in the sediment obtained after filtration (Kowalski & Makara, 2012; Polish patent application, 2012). The figures of treatment and filtration process of pig manure are shown in Table 2. The results of filtrate and after filtration sediment analyses are given in Table 3.

The consumption figures of phosphoric and sulfuric acids and lime milk used for the manure mineralization varied depending on the chemical composition of the manure. The higher dry matter content in the manure resulted in increased consumption of acids and lime milk.

Despite strong variations in COD (10 000–80 000 mg/L), in SS (10 000–20 000 mg/L), in TKN (2000–3800 mg/L) and in P_{tot} (500–900 mg/L), the method made it possible to maintain an overall average pollutant removal performance about 90% for the COD, >99% for the SS, to 47% for the TKN. The mineralization process eliminates also over 75% of the odor intensity coming from the obtained filtrate in comparison to odor intensity coming from the pig manure (Kowalski *et al.*, 2012; Sówka *et al.*, 2013).

Table 3. Results of filtrate and after filtration sediments analyses.

Determined parameter (mg/L)	Manure batch							
	I	II	III	IV	V	VI	VII	VIII
	Filtrate							
TKN	3012	2660	4200	6800	3230	5410	2100	2264
COD	2620	6000	4300	12200	3900	10700	1150	1900
Reduction in the filtrate (%): Concentration of COD	86.9	88.8	85.5	86.1	89.4	89.1	86.9	87.3
Load of COD	90.1	89.4	90.6	90.8	91.2	90.9	90.1	90.4
Reduction in the filtrate (%): Concentration of N	18.1	39.1	22.1	22.1	18.4	32.1	29.0	29.9
Load of N	38.10	42.65	49.61	48.22	32.30	43.37	46.35	47.27
SS	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
P_{tot}	5180	4818	3370	9130	3920	11330	1568	1347
	Filtered sediment							
C	12.18	15.61	5.00	16.97	10.78	13.26	13.56	4.63
H	2.22	2.68	1.33	2.86	2.05	2.3	2.33	1.16
N	0.91	1.15	1.08	1.66	0.93	1.23	1.18	0.68
Ca	22.17	25.33	29.75	18.72	23.42	19.68	21.49	32.59
K	0.19	0.39	0.64	0.47	0.19	0.29	0.21	0.29
Mg	0.38	0.78	0.31	0.55	0.50	0.52	0.54	0.38
P	11.45	13.10	13.34	7.98	10.96	8.05	10.05	14.95
S	1.41	2.01	1.38	2.27	1.57	1.50	1.53	1.07
Moisture content (%)	48.19	50.96	60.5	55.57	43.00	48.46	53.17	52.46

Treated filtrate could be used for artificial rain irrigation of crops or eventually as raw material for production of liquid fertilizer. If required, the filtrate may also be treated in conventional biological WWTP's. The increased P_{tot} content in the filtrate, 5–6 times higher than in pig manure is a problem. The goal of our further research is to decrease the phosphorus content in the filtrate.

The conducted operations resulted in crystallization of inorganic compounds in the slurry. Their presence would help to improve the filtered sludge structure and consequently to increase the filtration rate of treated manure slurry. The results of the semi-quantitative X-ray analysis of the phase content in the after filtration sediments presented in Table 4 showed that the content of amorphous substances in the samples is rather high reaching, in some cases even about 60% of sample mass. The crystalline phase whose content was equal to 60% of the mass of dried sample contained different calcium phosphates mostly hydroxyapatite. In some samples calcium sulfates and small amounts of silica were determined.

Table 4. Results of the semi-quantitative X-ray analysis of the phase content in the after filtration sediments (denotation of samples as in Table 3).

Sample	Content (%)							
	$Ca_5(PO_4)_3(OH)$	$CaHPO_4$	SiO_2	$CaHPO_4 \cdot 2H_2O$	$CaSO_4 \cdot 0.5H_2O$	$Ca_2H_2P_2O_7$	$CaSO_4 \cdot 2H_2O$	Amorphous phase
I	44.40±3.30			6.92±1.26		2.25±1.15	3.58±0.96	42.85±3.9
II	45.8±3.90			5.34±1.14			4.35±0.75	44.51±4.5
III	36.93±2.94	10.5±2.20						52.57±3.3
IV	28.18±2.67	11.5±4.20						60.32±4.5
V	38.5±3,00		0.88±0.63	12.62±2.16	2.90±1.15			45.1± 4.5
VI	31.1±3.20			4.39±0.96	3.20±0,52	2.50±1.1		58.81±4.8
VII	48.0±4.60		0.19±0.28	9.30±1.44	3.61±0.41			38.9±6.0
VIII	50.30±4.30	6.02±2.19						43.68±3.9

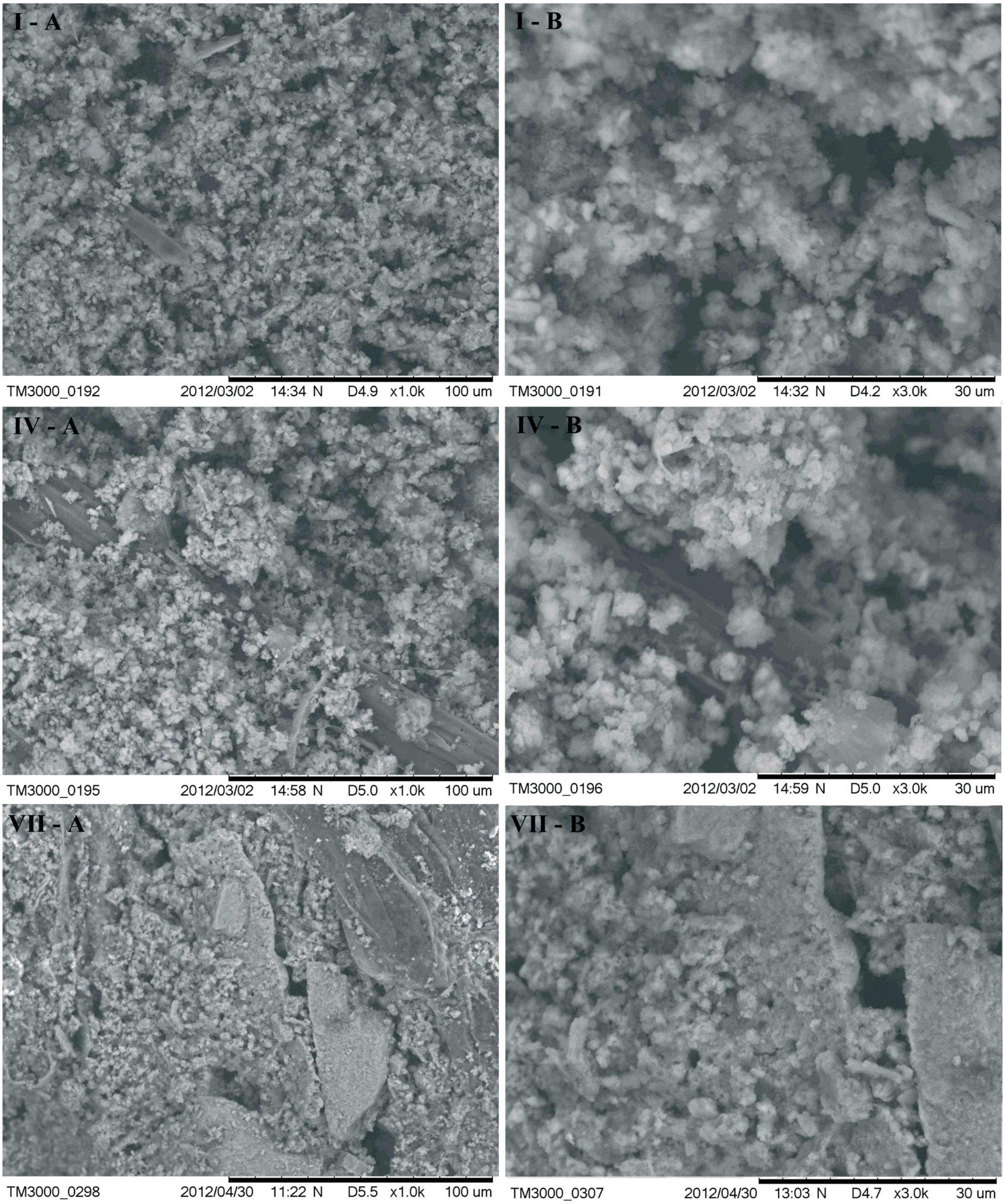

Figure 1. SEM images of the dried sediments (samples I, IV and VII — magnification A — 1000 B — 3000).

SEM images confirmed the presence of a considerable amount of minor crystallites (Fig. 1).

Thermal analyses of the after filtrate sediments (Fig. 2) showed that in time of samples heating in the temperature range to 70°C take place elimination of absorbed water and next in 150°C decomposition of calcium hydrates present in the samples. Thermal effects connected with decomposition of organic compounds and transformation of calcium phosphates were observed at 350–500°C. Two well-marked exothermic effects were to be seen at temperatures about 350°C (more intensive) and 460–470°C. Minor thermal effects observed at >600°C are connected probably with decomposition of calcium phosphates.

The results of X-ray and SEM analyses of the filtrate sediments confirmed that the worked out process of the pig manure mineralization allowed to incorporate into solid phase of slurry about 40–60% of crystalline phase (contained mostly calcium phosphates) that constituted some type of filtration aid, enabled achieving the high effectiveness of the filtration process. Simultaneously the used pig manure treatment process allowed coagulating considerable amount of organic phase contained micro particles that were incorporated into sediment. It re-

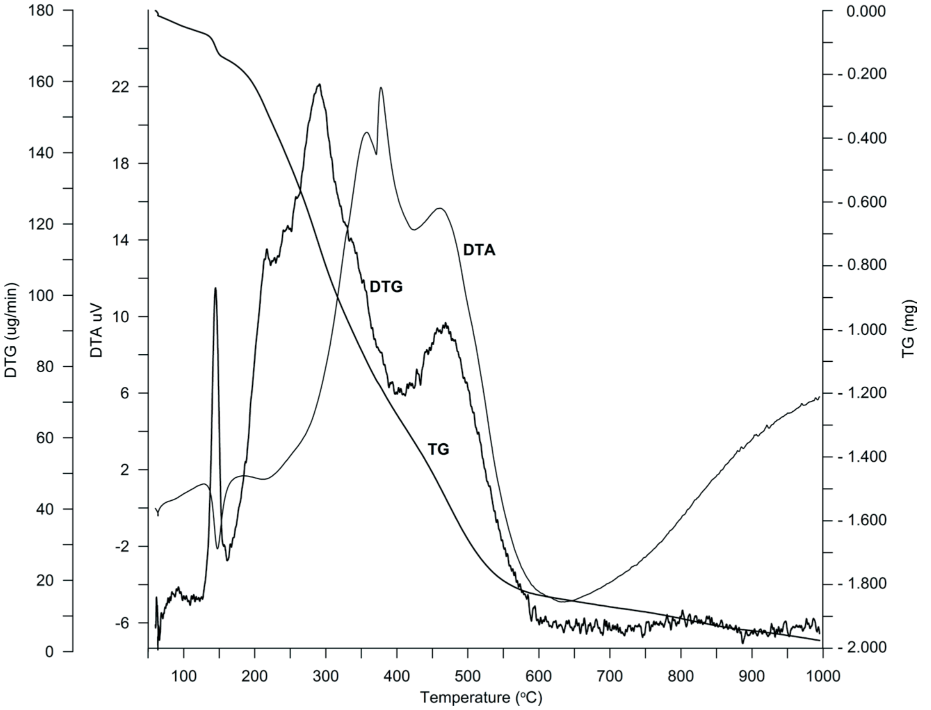

Figure 2. Thermal analyses of the sediment — sample I (denotation as in Table 3).

sulted in very high (about 90%) COD decrease in the filtrate in comparison to its content in the pig manure.

The sediment containing high quantities of bio-available calcium phosphates (Kowalski *et al.*, 2012) will be used as a semi-product in order to obtain mineral - organic fertilizer containing nitrogen, phosphorus, potassium, calcium and sulfur compounds, and microelements of universal nature (Hoffmann *et al.*, 2013).

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