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## THE QUALITY OF SEWAGE SLUDGE STABILIZED FOR A LONG TIME IN REED BASINS

The aim of the paper was to determine changes of sewage sludge quality stabilized for a long in reed basins (from 7 to 15 years). The following parameters were evaluated: dry matter and organic matter concentration, nitrogen, phosphorus, and selected heavy metal (Cd, Ni, Pb, Cr, Cu, Zn) fractions. The material for the investigations was collected from 4 reed basin systems (utilisation of sewage sludge) located in conventional WWTPs in Denmark (serving from 9 000 to 40 000 person equivalent, producing from 232 to 870 t of d.m/a). Based on the results obtained, it was found that the sludge in reed basins was dewatered and stabilized. Furthermore, it was found that the concentration of nitrogen decreased while the concentration of phosphorus increased. The analyzed heavy metals were mostly bound with the residual fraction and their concentrations were below permissible values for agricultural usage as determined by Polish law. Thus the product obtained – stabilized sewage-sludge, can be potentially used as fertilizer.

### 1. INTRODUCTION

The hydrophyte method utilizes wetland plants, i.e. *Phragmites* (reed), *Typha* (cat-tails) and *Salix* (willow). These plants grow on mineral subsoil with overlying layers of sludge. In this method, reeds are used most often [1]. Hydrophyte systems are built as concrete constructions (beds) or as tight tanks placed in the ground (basins) [2].

The dewatering efficiency of sewage sludge in reed beds is comparable to mechanical equipment such as a filter press (concentrations of dry matter can even reach up to 40%) [3]. Additionally, the cost of dewatering 1 ton of sludge in the reed bed is lower, specifically only 5–10% of the overall cost using traditional sludge handling methods [1].

Reed systems are especially useful in rural areas and housing estates where economical considerations limit the use of expensive mechanical equipment. Reed bed drying systems can be established in any area and are simple to build and service.

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Their low energy consumption is their main advantage [2].

Based on long-term experience, Danish companies (Hedelskabet Company and Orbicon) connected with the operation of hydrophyte systems (NIELSEN [1], [3]) recommended reed systems be built using several basins, namely at least 8. This makes it possible to supply raw sludge (irrigation) and ensure the time of rest (without irrigation). The average time of system operation is 10 years, although some systems can be operated much longer, even 15 years, without emptying [4].

It has been proven that dewatering sewage sludge in reed basins results in stabilized, sanitary sludge [3]. After this process, sludge from reed basins has a chemical composition similar to that of humus; therefore, it is possible to use it as a fertilizer in agriculture. Additionally, it was proven that the product obtained is safe as microbiological regards [5]. Heavy metals accumulate in sewage sludge and can be toxic for the environment. Taking account of an environmental protection, it is important to determine not only total concentrations of heavy metals in sewage sludge, but also their bound fractions. Heavy metals can have different fractions which influence their release into environment.

For completing the pilot study and due to a lack of hydrophyte system operation in Poland, the work was completed in cooperation with the Danish companies Hedelskabet and Orbicon, which made it possible to carry out the investigations in reed systems already operating for a long time in WWTPs. The aim of this research was to evaluate sludge that accumulated in reed basins for 7–15 years for changes in dry and organic matter, nitrogen and phosphorus concentrations, as well as the concentrations of heavy metals: Cd, Cr, Cu, Ni, Pb and Zn.

## 2. RESEARCH PROCEDURES

Secondary sludge stabilized in reed basins was used for testing. Samples of sludge were collected from 4 so-called Rodman basins. The basins were located in conventional WWTPs in the following Danish cities: Rudkøbing, Naskov, Vallo and Helsingør. The results from the locations analyzed are presented in table 1.

Table 1

Characteristics of the sites analyzed

Location of sites	Time of operation (years)	Number of basins	Total area (m <sup>2</sup> )	Amount of sludge (t d.m.·year <sup>-1</sup> )	pe*
Rudkøbing	13	8	5 000	232	13 000
Naskov	15	10	4 700	870	33 000
Vallo	7	8	3 867	300	9 000
Helsingør	9	10	10 500	630	40 000

pe – person equivalent.

In Rudkobing, raw sludge was not supplied to the reed basin for 2 years before sample collections. To object in Vallo raw sludge was supplied for 3 months and in Naskov for 2 months before sample collections. While in Helsingø, raw sludge was supplied for merely 2 weeks before sample collections.

The samples analyzed were collected at 6 points evenly spaced along the axis of symmetry for each basin. Samples of sludge from the same layers were averaged. The samples were collected by specially prepared equipment.

The dry matter in sludge was determined as the remainder after drying at a temperature of 105 °C to a constant weight according to the guidelines PN-78/C-04541 [6].

The organic matter determination consisted in ashing, thus homogenizing the samples at a temperature of 450 °C for 8 hours. It was assumed that losses from ignition correspond to the share of organic matter in the samples.

Kjeldahl nitrogen, i.e. the sum of organic and ammonia nitrogen, was determined in the sludge analyzed. The sludge sample was dried and homogenized. It was then alkalized using a 35% solution of NaOH and mineralized in the presence of the catalyst  $\text{CuSO}_4 + \text{K}_2\text{SO}_4$  using ammonium distillation. Sample mineralization was completed using Digestion Systems 1006 from the Swedish Company Tecator. The determination of ammonia nitrogen was carried out using the distillation method in the Kjeltec System 1026 from the Tecator company.

For determining the phosphorus concentration, the sample was dried, homogenized, and then mineralized using a mixture of the concentrated acids  $\text{HClO}_4$  and  $\text{HNO}_3$ . In the solution obtained,  $\text{PO}_4^{3-}$  ions were determined colorimetrically (the reaction with ammonia molybdate in the presence of glycerin with dissolved  $\text{SnCl}_2$ ). Sample mineralization was completed in the Digestion System 1006 manufactured by the Swedish company Tecator. Colorimetric measurements were carried out in the Aquatec 5400 – Analyzer from the company Tecator.

The determination of individual fractions of heavy metals was completed using the sequential extraction method according to The Community Bureau of Reference – BCR [7]. The test used 4 solutions: 0.11 molar  $\text{CH}_3\text{COOH}$  (Fraction I), 0.05 molar  $\text{NH}_2\text{OH}\cdot\text{HCl}$ ; pH 2 (Fraction II), 1 molar  $\text{NH}_3\text{COONH}_3$ ; pH 2 (Fraction III), and mixture of HF and  $\text{HNO}_3$  (Fraction IV). Using these extractions, the following four fractions of metals were separated: ion exchange and carbonate fraction (I – the most mobile), hydroxide fraction (bounded with oxide and hydroxide iron and magnesium) (II), organic fraction (III), residual fraction (IV – the most stable).

### 3. RESULTS

Based on the results obtained, it was found that the dry matter concentration in sludge stabilized in reed basins reached 36.9%. The increase in the dry matter concentration after long-term stabilization was from 26.5% to 41.3%. Table 2 presents

changes of dry matter concentration in layers of stabilized sludge from the samples analyzed.

Table 2

Changes in dry matter concentration of sewage sludge during stabilization in reed basins (in %)

Location	Vallo	Helsinge	Rudkobing	Naskov
Time of stabilization (years)	7	9	13	15
Before stabilization	21.6±0.7	14.9±0.4	26.4±0.4	20.2±0.4
After stabilization	29.4±1.6	25.4±1.9	36.9±1.7	29.7±0.5
Increase of dry matter concentration	26.5	41.3	28.5	32.0

Table 3 presents changes of organic matter concentration in sewage sludge during its long-term stabilization in reed basins. It was found that reed basins resulted in a decrease in the organic matter concentration, on average from 47.1 to 39.2% of d.m.

Table 3

Changes of organic matter concentration in sewage sludge stabilized in reed basins (in % of d.m.)

Location	Vallo	Helsinge	Rudkobing	Naskov
Time of stabilization (years)	7	9	13	15
Before stabilization	51.4±0.5	46.7±0.5	43.5±0.2	46.9±0.8
After stabilization	40.4±0.6	38.0±0.3	36.6±0.3	41.7±0.7
Decrease of organic matter concentration	21.4	18.6	15.9	11.1

The changes in the Kjeldahl nitrogen concentration in sewage sludge stabilized in reed basins are presented in table 4. In this case, a nitrogen reduction, in the range from 18.2 to 34.8%, was observed in the time of stabilization.

Table 4

Changes in the Kjeldahl nitrogen concentration in reed basins after stabilization (in % of d.m.)

Location	Vallo	Helsinge	Rudkobing	Naskov
Time of stabilization (years)	7	9	13	15
Before stabilization	2.7±0.2	2.2±0.1	2.3±0.2	2.8±0.1
After stabilization	1.9±0.2	1.8±0.1	1.5±0.1	2.0±0.1
Decrease of Kjeldahl nitrogen concentration	29.6	18.2	34.8	28.6

Changes in the phosphorus concentration in sludge stabilized in reed basins are presented in table 5. An increase in the phosphorus concentration from 1.1 to 31.0% was obtained during stabilization.

Table 5

Changes in the phosphorus concentration in sewage sludge stabilized in reed basins (in % of d.m.)

Location	Vallo	Helsinge	Rudkobing	Naskov
Time of stabilization (years)	7	9	13	15
Before stabilization	3.56±0.2	3.49±0.2	4.58±0.2	3.65±0.2
After stabilization	5.16±0.3	3.94±0.2	4.63±0.5	4.76±0.2
Increase of phosphorus concentration	31.0	11.4	1.1	23.3

Table 6

Average concentrations of heavy metals and their speciation for sludge stabilized in reed basins

Location	Average concentrations (mg·kg d.m. <sup>-1</sup> )	Permissible values for agricultural use (mg·kg d.m. <sup>-1</sup> )	Fraction (%)			
			I	II	III	IV
Cd						
Vallo	1.07±0.13	10	n.d.*	8.4	16.8	74.8
Helsinge	0.95±0.12		n.d.*	23.2	23.2	53.6
Rudkobing	0.84±0.17		n.d.*	26.2	15.5	58.3
Naskov	0.74±0.06		n.d.*	n.d.*	27.0	73.0
Pb						
Vallo	10.0 ± 1.8	500	n.d.*	n.d.*	5.1	94.9
Helsinge	10.7 ± 3.6		n.d.*	n.d.*	10.1	89.9
Rudkobing	14.6 ± 2.7		n.d.*	n.d.*	7.1	92.9
Naskov	15.6 ± 2.3		n.d.*	n.d.*	8.0	92.0
Cr						
Vallo	11.3 ± 1.2	500	n.d.*	n.d.*	10.9	89.1
Helsinge	18.1 ± 4.5		n.d.*	n.d.*	10.2	89.8
Rudkobing	32.1 ± 5.7		n.d.*	n.d.*	16.0	84.0
Naskov	17.5 ± 5.1		n.d.*	n.d.*	4.2	95.8
Ni						
Vallo	26.3 ± 1.6	100	6.2	3.6	15.0	75.2
Helsinge	20.7 ± 2.7		10.2	13.4	15.3	61.1
Rudkobing	20.3 ± 3.4		9.7	10.7	13.4	66.2
Naskov	22.4 ± 2.1		8.3	4.1	23.9	63.7
Cu						
Vallo	165.6 ± 10.3	800	1.3	0.2	16.3	82.2
Helsinge	236.6 ± 41.5		2.4	0.4	35.1	62.1
Rudkobing	218.6 ± 54.3		1.8	0.4	17.1	80.7
Naskov	80.8 ± 10.6		2.9	0.6	29.7	66.8
Zn						
Vallo	520.2 ± 43.5	2500	3.5	9.7	22.6	64.2
Helsinge	416.0 ± 57.6		7.6	27.3	39.4	25.7
Rudkobing	542.2 ± 95.6		8.3	26.1	19.2	46.4
Naskov	437.1 ± 39.1		6.7	15.1	37.7	40.5

n.d. – not detected.

Table 6 presents average concentrations of the heavy metals analyzed as well as their speciation in sludge stabilized in reed basins.

#### 4. DISCUSSION OF RESULTS

For the sewage sludge stabilized in reed basins, the lowest concentrations of dry matter were in layers before the process, while the highest concentrations of dry matter were in the layers after long-term stabilization (table 2). The decrease in moisture was caused by the sludge forming aggregate particles. This decrease was caused by transpiration as well as gravitational outflow of water aided by roots and rhizomes. Differences in the dry matter concentration depended on the time of sludge dewatering. The system with the highest concentration of dry matter (Rudkobing) was not supplied with raw sewage sludge for two years before sample collection. The system with the lowest concentration of dry matter, located in Helsingø, was supplied with raw sludge just 2 weeks before sample collection.

Conversely, as in the case of dry matter, the lowest concentrations of organic matter were found in layers of sludge before stabilization, while the highest concentrations of organic matter were in the layers after stabilization (table 3). Notably, the average concentration of organic matter in sludge treated in the pioneer reed bed in Darżlubie, Poland, was similar to those obtained in the tested samples, averaging from  $50.1 \pm 4.3\%$  of d.m. in layers before stabilization to  $43.3 \pm 4.2\%$  of d.m. in layers after stabilization [8]. Based on these results, it is likely that the organic matter in the sludge was biodegraded and stabilized. Similar conclusions were reached in research conducted by NIELSEN [1] and OBARSKA-PEMPKOWIAK and SOBOCIŃSKI [2].

According to Metcalf's & Eddy's [9] findings, the average concentrations of nitrogen in sewage sludge changed from 0.5 to 7% of d.m. The average values of the nitrogen concentration in the sewage sludge analyzed were within this range [9]. They were also similar to the average values reported by ZWARA & OBARSKA-PEMPKOWIAK [8] for sewage sludge dewatered in reed bed in Darżlubie. Based on the investigations, Kjeldahl nitrogen concentrations were found to decrease, on average from 2.5 to 1.8% of d.m. (table 4). This is caused by microbiological processes of nitrification and denitrification as described by NIELSEN [3].

According to METCALF & EDDY [9], the average concentration of phosphorus compounds in sewage sludge changed from 0 to 3% of d.m. The average values obtained for the phosphorus concentration in the sludge analyzed were clearly higher. Compared to the average values obtained by ZWARA and OBARSKA-PEMPKOWIAK [8], phosphorus concentrations in the sludge analyzed were almost 10 times higher (table 5). This significant difference could be caused by a higher consumption of detergents in Denmark as well as an almost three times longer sludge stabilization time for the analyzed samples. The results obtained confirm that long-term stabilization of sewage sludge causes an in-

crease in the phosphorus concentration, which is probably due to decomposition of organic matter in addition to a decrease in volume of stabilized sewage sludge.

The research showed that sewage sludge stabilized in reed basins can be used in agriculture since the heavy metal concentrations are below permissible legal values specified in the regulations of the Polish Ministry of the Environment (table 6) [10].

Heavy metals determined in sludge were mostly bound with the most stable combination (fraction IV). Based on this, it can be concluded that long-term sludge stabilization in reed basins produces a safe product. Chromium and lead were the most stable metals with their share of the residual fraction being about 90%. The reference data for sludge stabilized by conventional methods confirms that the highest share of lead is a part of fraction IV (SCANCAR et al. [7] and ÁLVAREZ et al. [11]). According to these authors, chromium and copper were mostly bound with fraction III. In the sludge analyzed, zinc was the most mobile metal with its share of I, II, III fractions being the highest (table 6). Also the authors of [11], [12] confirm that fractions II and III prevail in sludge stabilized by conventional methods. According to the data published [7], [11], [12] in conventionally stabilized sludge, cadmium and nickel were mostly bound with mobile fractions.

The samples with the highest concentration of metals bound with mobile fractions were located in Helsinge. These particular samples were supplied with raw sludge merely 2 weeks before sample collection. Thus, it can be concluded that the recently supplied raw sludge caused the increase in heavy metal concentration bound with the mobile fractions.

## 5. CONCLUSION

Based on the results of research concerning sewage sludge quality during its long-term stabilization in reed basins, the following conclusions were formulated:

1. Sludge stabilization resulted in an increase in the dry matter concentration and a decrease in the organic matter concentration for all the samples tested.
2. It was observed that phosphorus concentration increased by about 1.1–31.0%, while Kjeldahl nitrogen concentration decreased by about 18.2–34.8%.
3. The concentrations of heavy metals in stabilized sewage sludge were below obligatory values for agricultural use.
4. Heavy metals were mostly bound with the most stable residual fraction.
5. The stabilized sewage sludge can potentially be used as fertilizer due to the relatively low concentration of heavy metals and their presence in stable fractions, the presence of nitrogen compounds, and high concentrations of phosphorus.

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#### JAKOŚĆ OSADÓW ŚCIEKOWYCH STABILIZOWANYCH DŁUGOOKRESOWO W BASENACH TRZCINOWYCH

Analizowano zmiany jakości osadów ściekowych stabilizowanych długookresowo w basenach trzciniowych (w okresie 7–15 lat). W tym celu oznaczono stężenia suchej masy, substancji organicznej, azotu i fosforu oraz całkowite stężenia i dokonano specjacji wybranych metali ciężkich (Cd, Ni, Pb, Cr, Cu, Zn). Materiał badawczy pobrano z czterech trzciniowych systemów utylizacji osadów ściekowych znajdujących się na terenie konwencjonalnych oczyszczalni w Danii (zasilanych ściekami od 9000 do 40000 mieszkańców, wytwarzających od 323 do 870 t s.m. osadów nadmiernych/rok). Na podstawie uzyskanych wyników stwierdzono, że w basenach trzciniowych osady ulegały odwodnieniu i stabilizacji. Wykazano, że stężenia azotu malały, podczas gdy fosforu – wzrastały. Stwierdzono również, że analizowane metale ciężkie były związane przede wszystkim z frakcją rezydualną, a ich stężenia były niższe od wartości dopuszczalnych określonych przez polskie przepisy odnoszące się do rolniczego wykorzystania osadów. Uzyskany produkt, tj. ustabilizowane osady ściekowe, może być potencjalnie traktowany jako substancja nawozowa do wykorzystania w rolnictwie.