Int. J. Environ. Sci. Technol. (2016) 13:1607–1614
DOI 10.1007/s13762-016-0991-6

SHORT COMMUNICATION

Appraisal of changes in sewage sludge management

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Received: 9 June 2015 / Revised: 2 December 2015 / Accepted: 12 April 2016 / Published online: 27 April 2016

Abstract A positive energy balance caused by accelerated production of CH_4 and additional financial benefits associated with shorter retention times, reduction in volumes, easier dewatering, enhanced hygiene, etc., are the reasons why most modern wastewater treatment plants are newly equipped with hydrothermal pretreatment units. This study examined how the changes in composition of the pretreated sewage sludge fermentation residues manifested themselves in subsequent processing by pyrolysis, both in terms of technology and financial impacts. It has been found that concentrations of heavy metals and other hazardous inhibitors might increase; however, the overall biotoxicity is lower due to their immobilization in the charred mass. Moreover, charring of the fermentation residues results in significant financial as well as technological benefits.

Keywords Sewage sludge management · Hydrothermal decomposition · Cost analysis · Environmental policy

Introduction

Sewage sludge (hereinafter also SS) consists mainly of microbial cells that commonly withstand enzymatic attacks and water. Numerous reviews and research studies of sewage sludge management have been published recently (Kelessidis and Stasinakis 2012). This is undoubtedly due to the fact that sewage sludge management is a growing problem, especially in highly populated areas (Samaras et al. 2014). The highest attention is paid to advantages associated with hydrothermal pretreatment (hereinafter also HP) which is a combination of higher temperatures and pressurized water as a reaction medium (Yin et al. 2015). Serrano et al. (2015) confirm that the optimum process parameters (also hydraulic retention times, hydromodule, etc.) of hydrothermal pretreatment were found—by interpolation of results obtained during numerous simulations (Xue et al. 2015) and subsequent multifactor optimizations (Maroušek 2012)—in the proximity 160–180 °C and 15–120 min (Donoso-Bravo et al. 2010). If the processing parameters are severe enough for a sufficient time, the cell walls may be destroyed, making the proteins easily accessible to biological degradation (Ruiz-Hernando et al. 2013). Reuse of waste heat (Speidel et al. 2015) from combustion of biogas released in subsequent anaerobic fermentation (Reza et al. 2014) makes the energy balance of the hydrothermal pretreatment positive and therefore financially attractive (Ruffino et al. 2015). This practice, which has been transferred into the commercial sector only recently, results in many technological and economical advantages (Wirth et al. 2015). Faster release of easily biodegradable organic matter means savings from mass and volume reduction (Maroušek 2013) and represents new financial opportunities linked with easier dewatering (Ruiz-Hernando et al. 2013), stabilization and byproduct recycling (Strong et al. 2011). In particular, reuse of phosphorus (Nanzer et al. 2014) is the main reason why fermentation residues from sewage sludge are still used for agronomical purposes in some developing countries. In developed countries, due to higher concentrations of microorganisms and harmful substances, such as heavy metals, poorly biodegradable

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organic compounds, bacteria, viruses, pharmaceuticals, hormones and dioxins, the sewage sludge fermentation residues are routinely processed by combustion or pyrolysis (Magdziarz and Werle 2014). Numerous trials were performed to map kinetics of hydrothermal pretreatment of sewage sludge in detail and to develop a new generation of devices (Abelleira et al. 2012) which allow multiplication of effectiveness of the subsequent anaerobic fermentation. Hydrothermal pretreatment is now used before anaerobic fermentation in a commercial scale worldwide as a technologically, as well as economically, promising technology in SS management to achieve not only the above-indicated benefits but also for better removal of pathogens, odor control and removal of toxic compounds and for human health and environmental protection (Kelessidis and Stasinakis 2012).

A hypothesis was formulated to determine whether or how the better removal of organic matter manifests itself in quality indicators of the fermentation residues (Váchalová et al. 2014a). The hypothesis further investigated whether the effect can be further increased by a subsequent pyrolysis procedure. How does this practice change qualitative indicators of the ash and solid pyrolysis residues? No satisfactory answers to such questions have been found in the literature. However, this topic is highly significant because hydrothermal pretreatment has been a part of the design of nearly all new sewage water treatment units and also a part of refurbishment of the existing facilities.

Materials and methods

To assess the overall technological and also economical effects, two sewage sludge samples (with and without HP) were exposed to anaerobic fermentation at identical processing parameters to obtain different fermentation residues (Fig. 1). Mechanically cleaned fresh SS after decantation (Table 1) was obtained from the wastewater treatment plant in České Budějovice (ČEVAK a.s., Czech Republic). Volatile solids (hereinafter VS, %) were analyzed using the EW 220-3NM electronic weight scales (KERN & SOHN GmbH, Germany) and Vacucell laboratory dry heat vacuum oven (BMT, USA) by drying at 95 °C to a constant weight ($n = 6$, $\alpha < 0.01$). Chemical oxygen demand was determined by potassium dichromate (hereinafter COD_{Cr}, g L^{-1}) according to Raposo et al. (2010). Dissolved volatile fatty acids (hereinafter ΣVFA, g L^{-1}), pH, Total Kjeldahl Nitrogen (hereinafter TKN, mg L^{-1}) and other routine indicators were established according to routine laboratory procedures. With the assumed inaccuracy, the sum of proteins (further ΣPs, mg L^{-1}) was only estimated from a simplified assumption that the proteins contain 16 % of N. Prior to hydrothermal pretreatment, the sewage sludge was diluted with rainwater to 10 % VS and preheated to 75 °C to avoid pressure turbulences during the HP processing. The preheated and diluted sewage sludge was transferred by a high-pressure screw pump into the TTP4 HP technology (Biomass Technology a.s., Czech Republic) for hydrothermal treatment (in a thick-walled 400-L reactor equipped with a horizontally placed helix) at 1.1 MPa and 20 min of the hydraulic retention time (Fig. 2). An expansion tourniquet (located at the end of the main reactor) was set up to perform a single 0.3 L explosion in 0.1 s. No hydrolyzing agent was added. The sewage sludge sample exposed to the above-described hydrothermal pretreatment, as well as the blank sample (without HP), was subsequently processed in a battery of fully automatically monitored semi-batch 70 L reactors (Stix Ltd., Czech Republic) equipped with a GA3000 infrared-based biogas analyzer (Chromservis Ltd., Czech Republic) and other online sensors (Maroušek et al. 2012). The anaerobic fermentation (40 °C;

Table 1 Analysis of SS, the abbreviations and the corresponding units are provided in the "Technology and methods" section (n and α as in Fig. 2)

VS (%)	73
COD_{Cr}	151.33 ± 4.85
ΣVFA	2.01 ± 0.46
pH	7.27
TKN	6103.73 ± 86.34
ΣPs	46.00 ± 8.79

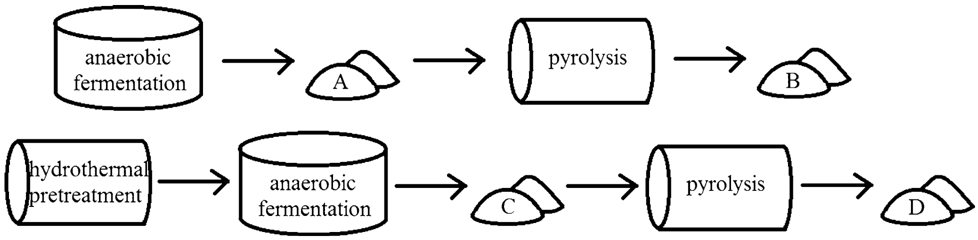

Fig. 1 Experimental setup, where: *A* fermentation residues of SS; *B* solid pyrolytic residue of *A*; *C* fermentation residue of SS that was previously exposed to hydrothermal pretreatment; *D* solid pyrolytic residue of *C*

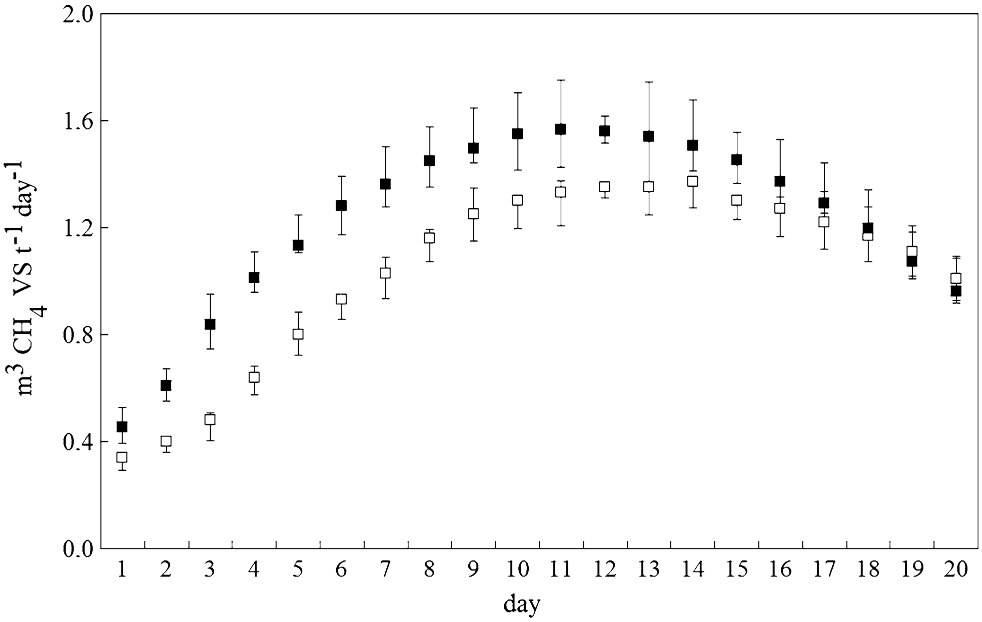

Fig. 2 The dynamics of daily CH_4 production (where *black squares* represent SS after HP and *empty squares* represent samples without pretreatment; the *bars* indicate the standard deviations $n = 6$; $\alpha = 0.05$). The diagram shows that HP may significantly accelerate biodegradation of SS

mild agitation) was interrupted after 20 days. The fermentation residues were analyzed for hydrolyzability (by H_2SO_4) according to Shirato and Yokozawa (2006) using an automatic highly sensitive N/C analyzer (NC-90A, Shimadzu), while the loss on the labile pool 2 of hydrolysable carbon was chosen as the main criterion (hereinafter LP2, %). The analyses of adsorbable organic halogens (hereinafter ΣAOX, mg kg^{-1}); polychlorinated biphenyls congeners 28, 52, 101, 138, 153 and 180 (hereinafter ΣPCB, mg kg^{-1}); polychlorinated dibenzodioxins and furans (hereinafter ΣPCDD/F, mg kg^{-1}); polybrominated diphenyl ethers (hereinafter ΣPBDE, μg kg^{-1}); nonylphenol and nonylphenol ethoxylates (hereinafter Σ(NP + NPEO), μg kg^{-1}); polycyclic aromatic hydrocarbons (hereinafter ΣPAH, mg kg^{-1}); di-(2-ethylhexyl) phthalate and dibutyl phthalate (hereinafter DEHP + DBP, mg kg^{-1}); sum of benzene, toluene, ethylbenzene and xylenes (hereinafter ΣBTEX); heavy metals As, Cd, Cr, Cu, Hg, Ni, Pb and Zn (mg kg^{-1}); extractable organic halogens (hereinafter ΣEOH, mg kg^{-1}); sum of aromatic hydrocarbon compounds containing between 10 and 40 carbon atoms (hereinafter $\Sigma C_{10} - C_{40}$, mg. kg^{-1}); acute toxicity for fish (*Poecilia reticulata*, hereinafter PR, %); daphnia immobilization (*Daphnia subspicatus*, hereinafter DAS, %); freshwater algae immobilization (*Desmodesmus subspicatus*, hereinafter DES, %); and rapeseed phytotoxicity (*Sinapis alba*, hereinafter SA, %), were performed according to routine laboratory methods in agreement with applicable EU standards. Subsequently, the fermentation residues were processed in the pK 02 (AIVOTEC, s.r.o., Czech Republic) continuous pyrolysis unit which consists of a vertical hopper and horizontal pyrolytic chamber equipped with a slowly rotating helix. The pyrolysis unit is designed to minimize production of other than solid pyrolytic residues, and it does not require any costly scrubber, and therefore, the main chamber is provided with an airtight hopper at the end to avoid spontaneous combustion of the resulting residues (Maroušek 2014). The pyrolytic residues were analyzed again with the above-described biochemical methods and additional qualitative indicators as provided below. The NOVA 4200e microporosity analyzer (Quantachrome Instruments, USA) was used to analyze single-point surface areas at $P/P_0 = 0.2$ (hereinafter P/P_0, m^2 g^{-1}), BET surface area (hereinafter BET, m^2 g^{-1}) and Langmuir surface area (hereinafter L, m^2 g^{-1}) by the N_2 adsorption method. The remaining analyses were carried out to European standards, routine laboratory practices or as described in previous publications by the authors. The JEOL 6700 analytical scanning electron microscope (JEOL, Japan) was used to observe morphology manifestations of the hydrothermal treatment and pyrolysis after coating the samples with white gold (Au + Pb) by a vacuum evaporation method using the JFC-1600 sputtering device (JEOL, Japan).

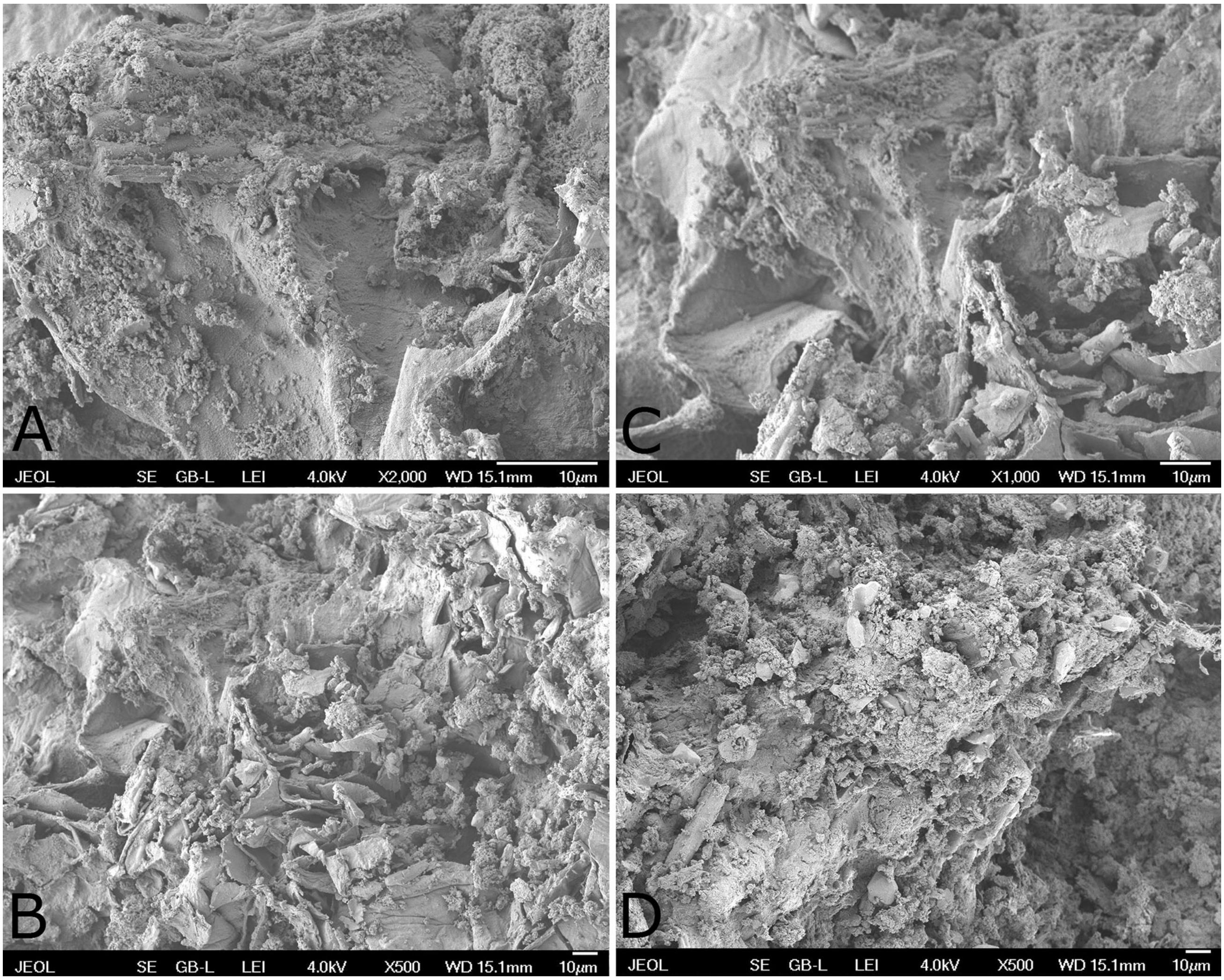

Fig. 3 Scans from the electron microscope allow observation of changes in the surface morphology of the samples (*A*–*D* as in Fig. 1) manifested as microporosity (P/P_0; BET and L, the abbreviations are explained in the "Technology and methods" section) by increase in the surface areas (see Table 2)

Detailed parameters of the microscope setup are described in the scans (Fig. 3).

Results and discussion

The European Urban Wastewater Treatment Directive (91/271/EEC) has lead to a significant increase in sewage sludge production and to strong limitations of its disposal routes (Ruiz-Hernando et al. 2013). Consequently, the research of efficient sludge treatments is essential to ensure proper management of sewage sludge and minimization of the costs. Table 1 suggests that the key indicators of the available sewage sludge samples were in the standard ranges (Xue et al. 2015) which provides good arguments for broader applicability of the obtained results (Donoso-Bravo et al. 2010). The obtained kinetics of CH_4 production (Fig. 1) does not match the best results presented in the literature (Cao and Pawłowski 2012), but it has confirmed the potential of hydrothermal pretreatment. The obtained results (24.695 in comparison with 20.810 m^3 CH_4 VS t^{-1}, i.e., an increase by 19 %) are more similar to those observed by Ruffino et al. (2015) who claims that the total revenues from sale of electricity increased by 10 % after the CH_4 yields increased by 21 %. The data have proved, among other things, that the operating parameters of the hydrothermal pretreatment were chosen correctly because once the temperature exceeds the recommended range, the Maillard reactions will occur to produce melanoidins as a result of reactions between reducing sugars and amino acids. However, melanoidins are heterogeneous polymers with high molecular weight and they are difficult to degrade or they even inhibit degradation of other organics (Dwyer et al. 2008). It can be assumed that faster CH_4 production could be achieved by increasing the operating temperature (Váchalová et al. 2014b), application of the inoculums or addition of easily fermentable organic matter (Maroušek et al. 2013) to improve (Serrano et al. 2015) the C to N ratio (Maroušek et al. 2014). However, in this case, more attention was paid to changes of composition of the fermentation residues (Table 2). Firstly, calorific value of fermentation residues after hydrothermal pretreatment increased by ca. 3 %. Given that the deviation associated with the calorimeter was almost 1 %, such a change can be hardly considered significant. On the other hand, calorific values of the pyrolyzed residues demonstrated a difference of 4 %. In the context of the methanogenic activity discussed above, this can be already considered a trend. It can be argued that this trend was confirmed by an analysis of removal of hydrolysable pools of organic matter, which was higher by one-third. Among other effects, hydrothermal pretreatment has also resulted in 14 % higher concentrations of ΣAOX in comparison with blank sample. However, the subsequent pyrolysis wiped the differences out. The levels of ΣPCB, ΣPCDD/F, Σ(NP + NPEO), ΣBTEX and ΣEOH were under the detection limits, and therefore, they cannot be discussed. The levels of ΣPAH, DEHP + DBP and $\Sigma C_{10} - C_{40}$ were low with negligible differences. Moreover, the concentrations were not found in the pyrolyzed residues. HP resulted in increased concentrations of heavy metals, both in the fermentation residues (on average by 8 %) and after the subsequent pyrolysis (on average by 14 %). Even after taking into account the measurement deviations, it should be noted that the concentration increases were not the same for all heavy metals. It would be interesting to further investigate this mechanism. It was found out that neither the raw sludge nor the pretreated SS was biotoxic to kill fish under normal concentrations. However, further results have revealed that HP pretreatment increases immobilization to daphnia (by 71 %) and algae (by 20 %). Increase in phytotoxicity to germination was also significant (by 50 %). By comparison, subsequent pyrolysis of the fermentation residues not only aligned but also decreased the biotoxicity factors. One could argue that this is inconsistent with elevated concentrations of heavy metals. However, some literature sources indicate that pyrolysis of the residues reduces bioavailability and phytotoxicity of heavy metals, especially Cd, Cu and Pb. Sequential fractionation of metals carried out by Park et al. (2011) indicated that biochar treatments substantially modified separation of Cd, Cu and Pb from the easily exchangeable phase to a less bioavailable organic-bound fraction. This is in good agreement with earlier findings by Uchimiya et al. (2010) who suggested that Cd, Cu, Ni and Pb immobilization during higher pyrolysis temperatures and activation lead to disappearance of certain functional groups (e.g., aliphatic –CH2 and –CH3) and formation of others (e.g., C–O) that are partly leachable. Immobilization of heavy metals resulted in non-stoichiometric release of protons, i.e., the total concentration of the immobilized metals was greater by several orders of magnitude than the protons released. The results suggest that with higher carbonized fractions and loading of chars the immobilization of heavy metals by cation exchange becomes increasingly outweighed by other controlling factors, such as coordination by π electrons (C=C) of carbon and precipitation. However, Table 2 indicates that heavy metals may not be the sole source of biotoxicity. The addition of solid pyrolysis residues not burdened by any of the discussed biotoxicity factors is a good starting point for application of the pyrolyzed fermentation residues into the soil. Processing by pyrolysis is advantageous also because of the big volume reduction, thermal destruction of toxic organics and recovery of the energy of organic sources in the sludge (Magdziarz and Werle 2014). Inguanzo et al. (2002) claims that, independently of pyrolysis conditions, all the solid products obtained from SS were highly macroporous, with relatively low meso- and micropore volumes. Microporosity analysis stated in Table 2 is in good agreement with those data. Lu et al. (1995) claimed that microporosity was reduced for chars obtained at 550–650 °C, possibly due to the pore enlargement phenomenon as a result of loss of volatiles in the intermediate thermoplastic phase. Data in Table 2 have also revealed that hydrothermal pretreatment may also have a synergic effect on enlargement of P/P_0, BET and L (+18, +73 and +63 %, respectively) surface area. The improvement of quality of the solid pyrolysis residue is another interesting finding from the economic point of view. Given that

Table 2 Analysis of the fermentation and pyrolysis residues (*A*–*D* as in Fig. 1) revealed many important differences

	A	B	C	D
HV	12.605 ± 0.072	12.965 ± 0.081	24.624 ± 0.066	25.622 ± 0.068
LP2	65	97	No data	No data
ΣAOX	207.15 ± 6.95	236.01 ± 8. 29	15.34 ± 5.07	15.73 ± 6.81
ΣPCB	No data	No data	No data	No data
ΣPCDD/F	No data	No data	No data	No data
ΣPBDE	8.15 ± 0.29	8.30 ± 0.15	No data	No data
Σ(NP + NPEO)	No data	No data	No data	No data
ΣPAH	2.4 ± 0.09	2.5 ± 0.12	No data	No data
DEHP + DBP	1.1 ± 0.07	1.1 ± 0.10	No data	No data
ΣBTEX	No data	No data	No data	No data
ΣEOH	No data	No data	No data	No data
$\Sigma C_{10} - C_{40}$	7.60 ± 0.87	7.91 ± 0.52	No data	No data
As	3.61 ± 0.19	3.72 ± 0.44	6.07 ± 0.47	6.11 ± 0.35
Cd	1.40 ± 0.07	1.42 ± 0.09	2.08 ± 0.33	3.42 ± 0.73
Cr	76.35 ± 5.61	90.43 ± 4.45	110.41 ± 8.89	108.63 ± 10.02
Cu	262.61 ± 8.17	307.35 ± 8.54	353.06 ± 11.49	355.72 ± 12.83
Hg	2.84 ± 0.43	2.88 ± 0.21	3.01 ± 0.20	3.02 ± 0.31
Ni	34.05 ± 3.80	39.45 ± 3.24	42.64 ± 4.05	55.36 ± 8.56
Pb	68.11 ± 1.52	69.54 ± 2.23	99.56 ± 7.17	113.65 ± 14.90
Zn	1304.46 ± 80.27	1393.95 ± 50.20	1583.10 ± 46.98	1609.05 ± 73.17
PR	0	0	0	0
DAS	7	12	5	5
DES	5	6	7	7
SA	4	8	3	5
P/P_0	0.23 ± 0.20	0.14 ± 0.11	5.63 ± 2.90	6.63 ± 1.76
BET	0.86 ± 0.52	1.06 ± 0.83	43.59 ± 6.58	75.32 ± 9.22
L	0.30 ± 0.25	0.15 ± 0.14	11.65 ± 4.43	19.04 ± 5.37

The abbreviations and the corresponding units are provided in the "Technology and methods" section (*n* and *α* as in Fig. 2). "No data" stands for concentrations under limit of detection

combustion standards (Mardoyan and Braun 2015) of the sewage sludge pyrolysis residues are met by mixing with charcoal that is free of pollutants and based on the obtained calorific values (Table 2), we can estimate its selling price as a fuel at ca. 150 $\text{EUR}_{\text{15th November 2015}}$ t^{-1}. Given that the acquisition cost of HP and pyrolysis technology is ca. 6 M $\text{EUR}_{\text{15th November 2015}}$ (for 30 t day^{-1}) and the running cost is 500EUR day^{-1}, we can expect the payback period to be slightly over 4 years. The project would be more attractive if the pyrolysis residue is sold for hobby farming or cultivation of ornamental plants. However, this would require high marketing expenses because farming with biochar is quite unknown to the public (Maroušek 2014).

Conclusion

It has been found out that hydrothermal pretreatment might significantly influence sewage sludge management, both in terms of technology and financial impacts. Proper disruption of the bacteria cell walls is manifested by higher removal of organic matters by roughly one-third. This is accompanied by accelerated CH_4 production and many advantages mentioned previously in the literature (shorter retention times, volume reduction, easier dewatering, enhanced hygiene, odor removal, etc.). For the first time, it has been proved that the fermentation residues consequently contain higher concentrations of hazardous elements and compounds. This has very dangerous implications in the application of these fermentation residues on arable land.

However, remedy was found and proved. The heavy metals may be successfully immobilized by the subsequent pyrolysis. The calculations have also shown that this technique might be relatively attractive from the financial point of view.

Acknowledgments The authors wish to extend their gratitude to all who supported this work.

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