



**Application of flocculated sewage  
sludge for growing miscanthus on post-  
mining lands**

**Mykola Kharytonov, Nadia Martynova, Mykhailo Babenko,  
Oleksandr Kovrov, Liliya Frolova & Paloma Hueso González**



# Application of flocculated sewage sludge for growing miscanthus on post-mining lands

Mykola Kharytonov <sup>a</sup>, Nadia Martynova<sup>b</sup>, Mykhailo Babenko<sup>a</sup>, Oleksandr Kovrov<sup>c</sup>, Liliya Frolova <sup>d</sup> and Paloma Hueso González<sup>e</sup>

<sup>a</sup>Agronomy Faculty, Dnipro State Agrarian and Economic University, Dnipro, Ukraine; <sup>b</sup>Botany Garden, Honchar Dnipro National University, Dnipro, Ukraine; <sup>c</sup>Department of Ecology and Technologies of Environmental Protection, Dnipro University of Technology, Dnipro, Ukraine; <sup>d</sup>Department of Inorganic Materials Technology and Ecology, Ukrainian State University of Chemical Technology, Dnipro, Ukraine; <sup>e</sup>Geomorphology and Soil Laboratory, Interuniversity Institute of Habitat, Territory and Digitalisation, University of Malaga, Malaga, Spain

## ABSTRACT

Miscanthus yields well on raw subsoil where organic matter and nutrients are supplied by sewage sludge; the yield is directly dependent on the amount of sludge applied; the greatest productivity (12.9t/ha) was achieved by adding 60t/ha of flocculated sludge. Among the macronutrients, nitrogen is most actively absorbed, the intensity of biomass accumulation of phosphorus and potassium is much lower; other essential elements can be ranked as Fe → Zn → Mn → Cu. Cobalt, nickel and cadmium do not accumulate in biomass but chromium does. The introduction of sewage sludge affects the thermal characteristics of the miscanthus biomass. The proportion of incombustible residue increases when sludge is applied at high doses. Application of 20t/ha flocculated sludge appears to be optimal for ensuring environmental safety and suitability as a fuel.

## KEYWORDS

Sewage sludge; loess-like loam; elemental accumulation

## Introduction

Soil is a complex system generating a number of interrelated functions and processes related to the geology and ecology of a landscape [1]. *Technosol* groups young soils developed from various man-made deposits including post-mining and dredged sediments at the beginning of fertility formation [2–5]. Crops grown in such infertile soils are usually limited by available nutrients. In the long term, biological reclamation is possible by growing hay with a legume-grass mixture [6] but, recently, reclamation assisted by application of some combination of wastes and industrial by-products has received increasing attention [7]. The compost is more effective in loamy as opposed to clayey materials [8]; on sandy soils, compost-biochar mixtures achieve a positive, synergistic effect on soil organic-matter and nutrient levels, and water-storage capacity [9].

The potential of marginal lands for second-generation bio-feedstock production has also received attention in recent years [10]. Where the natural soil profile has been lost,

exposing raw or contaminated soil parent material, the land may be used for fast-growing feedstock for bioenergy and high-value bio-based products [11–14]. In particular, miscanthus (*Miscanthus × giganteus*) can provide a variety of pellets, bioethanol or biogas [15], even on polluted, depleted and heavy soils, erosion-prone slopes, riparian buffer zones and groundwater protection areas [16–18].

Phytoremediation results show that miscanthus tolerates soil contamination with heavy metals [19,20]; there are several coefficients, bioconcentration/uptake indices that reflect nutrient and/or heavy metal migration from soil to plants [21,22]. Miscanthus rhizomes treated with growth-promoting bacteria accumulated minimal concentrations of Cu and Pb in their above-ground biomass, and some Zn accumulation in the leaves and stems was observed [23].

Miscanthus's modest first-year yield is a limitation [24] but application of sewage sludge quickly optimises plant nutrition [25]. Field experiments with reed canary grass and miscanthus in Poland showed that uptake of macronutrients increased along with increasing dose of sewage sludge in the range 10-20t/ha; uptake of heavy metals was also higher at the higher dose of sludge [26]. Miscanthus recovers N, P and K from sludge most effectively from lower rates of application [27] but intensive growth was reported from applications up to 40t/ha [28]. Further experiments showed that the yield-forming effects of sludge are comparable to mineral fertilisers [29]; the optimal fertiliser dose was 160kgN/ha, regardless of nitrogen source [30], yielding 19.8t dry biomass/ha. Miscanthus has a high calorific value and high cellulose content, comparable to wood rather than a grass [31,32]. Increasing levels of nitrogen fertilisation up to 202kgN/ha decreased the proportion of hemicellulose, acetyl groups, and ash [33] but increased volatile matter, heating value, and cellulose content of the harvested biomass [34]. The findings on plant composition and energy content indicated that sludge could be used effectively in increasing combustible feedstock without harmful gas emissions [35].

The objective of this research was to study the effects of flocculated sewage sludge on migration of macro- and microelements in a Technosol/miscanthus system and the processes of decomposition of the main components of the biomass.

## Materials and methods

The Nikopol manganese ore basin is made up of Neogene, Palaeogene, and Quaternary sediments [36]. Post-Pliocene sediments are covered by a continuous layer of microporous, calcareous loess and loess-like loams of Pleistocene age. Most of the worked-out mineral deposits have their overburden replaced without stratification; i.e. no topsoil is replaced during restoration.

The field experiment was established on phyto-meliorated loess-like loam, typical of the soil parent material of the steppes, at the Pokrov Research and Educational Station of Dnipro State Agrarian and Economic University at 47°39' N, 34°08' E, at an elevation of 60 m. Unmodified sewage sludge (SS) and sludge treated with DAMET polymer flocculant (SS+F) were used [37]. The polymer separates the solids by gravity and allows complete drying of the residual cake in a few months. The biosolids were applied at the outset of the experiment at 20, 40 and 60 tonnes of dry matter/ha and disked into the top 10–12 cm of the soil in the autumn of 2019. Miscanthus rhizomes were planted at

14 800 plants/ha in the spring of 2020. The biomass yield was estimated by weighing in the second year of the experiment.

One month after the sludge application, five soil samples were taken at a depth of 0–20 cm in each experimental plot and mixed using the envelope method, air dried to a constant weight, then passed through a 2 mm sieve to remove plant material and stones. Soil organic carbon was determined by dichromate oxidation [38], easily hydrolysed N following Cornfield [39]. Available phosphorus was estimated by extraction with sodium bicarbonate [40,41], exchangeable K by flame emission spectrophotometry [42]. According to Alina Kabata-Pendias [43], estimations of available trace elements in soils are based on extractions by various solutions: chelating agents (EDTA), buffered salts (NH<sub>4</sub>OAc), neutral salts (CaCl<sub>2</sub>, MgCl<sub>2</sub>, Sr(NO<sub>3</sub>)<sub>2</sub>, NH<sub>4</sub>NO<sub>3</sub>), and other extractants (like Coca Cola). We used ammonium-acetate buffer (pH 4.8) [21] shaken in closed flasks for 1 h and then filtered. Essential and toxic elements were determined by atomic absorption spectrophotometry (AAS) with a Saturn 3 (Ukraine) instrument.

The samples of biomass were dried in open air, then at 105 °C, cooled in a desiccator for 1 h and then weighed [23]. N was determined by Kjeldahl analysis [44]; total P concentrations by acid digestion [45], potassium by flame photometry. Oven-dried plant material was weighed into porcelain crucibles and ashed at 500°C in a muffle furnace. Metal analyses were performed by AAS, the ash being taken up in a suitable volume of 2 M HCl, depending on the amount of the trace elements present and their sensitivity in AAS [21]. Calibration was provided by national standard multi-component samples nos. 0243–2001, 0244 and 0246.

The modified bioconcentration factor (mBCF) was calculated as the ratio between metal concentration in the plant and mobile metal concentration in the soil [22]. A particular element uptake by the plants was calculated by multiplying its content and the plant's respective biomass [46]. The cited data represent the arithmetic means of three replicates of each sample.

Thermal analysis of dry miscanthus biomass was performed with a MOM Q-1500D Derivatograph-C (MOM, Budapest) following Paulik and others [47]. Differential mass loss and heating effects were recorded, and the results were processed with the software package supplied with the device. Samples of biomass were analysed dynamically at a heating rate of 10°C/min in air. The results obtained were processed by statistical methods at a significance level 95%.

## Results and discussion

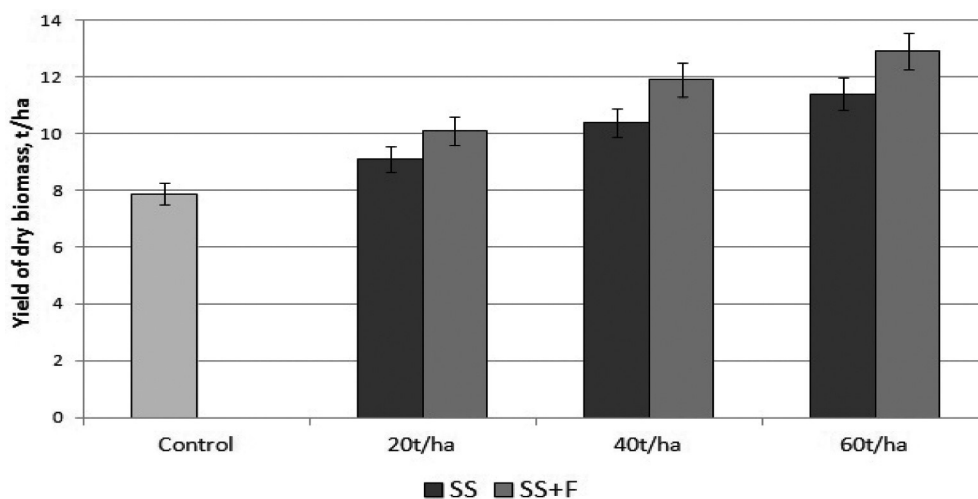
**Table 1** presents the main physical-chemical parameters of the phytomeliorated loess-like loam.

The content of easily hydrolysed nitrogen in the phytomeliorated substrate was well below the optimum level of 100 mg/kg but the flocculated sludge increased it 23–70%. Loess-like loam has a naturally high content of mobile phosphorus. The surface application of sewage sludge contributed up to 3–5 times more. The natural content of exchangeable potassium in loess-like loam is about average, and this was raised to the highest values in the plots receiving 60t/ha of sludge. The alkaline reaction of the

phytomeliorated loess-like loam was somewhat ameliorated by addition of sewage sludge.

**Table 1.** Characteristics of loess-like loam.

	Control	SS			SS+F		
		20 t/ha	40 t/ha	60 t/ha	20 t/ha	40 t/ha	60 t/ha
Humus (%)	1.19 ± 0.03	1.71 ± 0.06	1.86 ± 0.08	1.96 ± 0.08	1.60 ± 0.07	1.66 ± 0.06	2.14 ± 0.08
Dry matter (%)	96.6 ± 2.1	96.7 ± 1.9	96.9 ± 2.6	96.8 ± 2.5	97.2 ± 1.8	97.2 ± 1.9	97.0 ± 2.1
Total carbon (%)	0.69 ± 0.04	0.99 ± 0.06	1.08 ± 0.08	1.14 ± 0.09	0.93 ± 0.06	0.96 ± 0.05	1.24 ± 0.09
Easily hydrolysed nitrogen (mg/kg)	65.1 ± 1.8	80.0 ± 2.3	90.9 ± 2.5	103.8 ± 3.1	80.2 ± 2.3	92.2 ± 2.7	111.0 ± 3.6
Mobile phosphorus, P <sub>2</sub> O <sub>5</sub> (mg/kg)	42.2 ± 1.3	160.1 ± 2.6	188.1 ± 2.9	214.2 ± 3.1	139.0 ± 2.1	176.1 ± 2.6	225.7 ± 2.8
Exchangeable potassium, K <sub>2</sub> O (mg/kg)	206.5 ± 1.9	220.0 ± 2.5	226.6 ± 2.7	370.8 ± 3.8	227.2 ± 2.3	247.8 ± 2.5	350.2 ± 3.5
pH	8.35	8.3	8.2	8.1	8.25	8.1	8.0



**Figure 1.** Effect of sewage sludge on the yield of above-ground miscanthus biomass.

Two-year-old miscanthus grown in the control loess-like loam yielded a dry, above-ground biomass of 7.9t/ha. Application of sewage sludge, especially flocculated sludge, contributed to higher yields (Figure 1). For instance, the application of flocculated sewage sludge at 40t/ha yielded 11.9t/ha; 4.4% higher compared to adding unmodified sludge at a rate of 60t/ha. The highest yield, 12.9t/ha, was achieved with the introduction of flocculated sludge at 60t/ha.

Nitrogen accumulates intensively in the above-ground biomass of miscanthus. The introduction of sewage sludge contributed to an increase in nitrogen accumulation in

the biomass. The greatest effect was exerted by the use of SS+F in all treatments in comparison with SS (Figure 2).

Phosphorus and potassium accumulated in the biomass to a lesser extent. The application of sewage sludge at a dose of 60t/ha had the greatest effect on the concentration of phosphorus (Figure 3). With the accumulation of potassium, the use of unmodified sewage sludge was more effective at doses of 20 and 40t/ha than flocculated sewage sludge. At a dose of 60t/ha, both types of sludge had much the same effect.

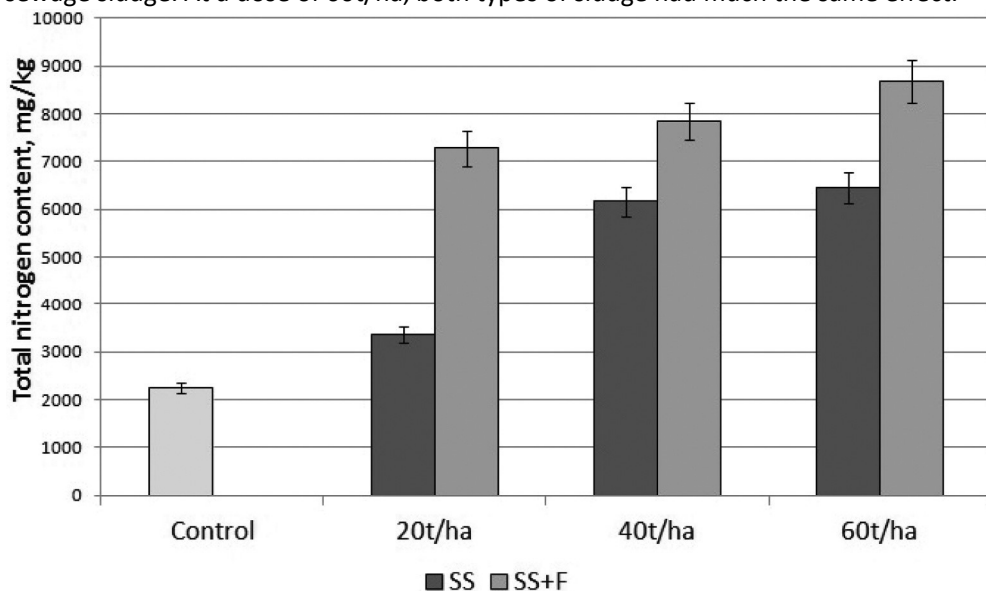


Figure 2. Nitrogen content in miscanthus biomass.

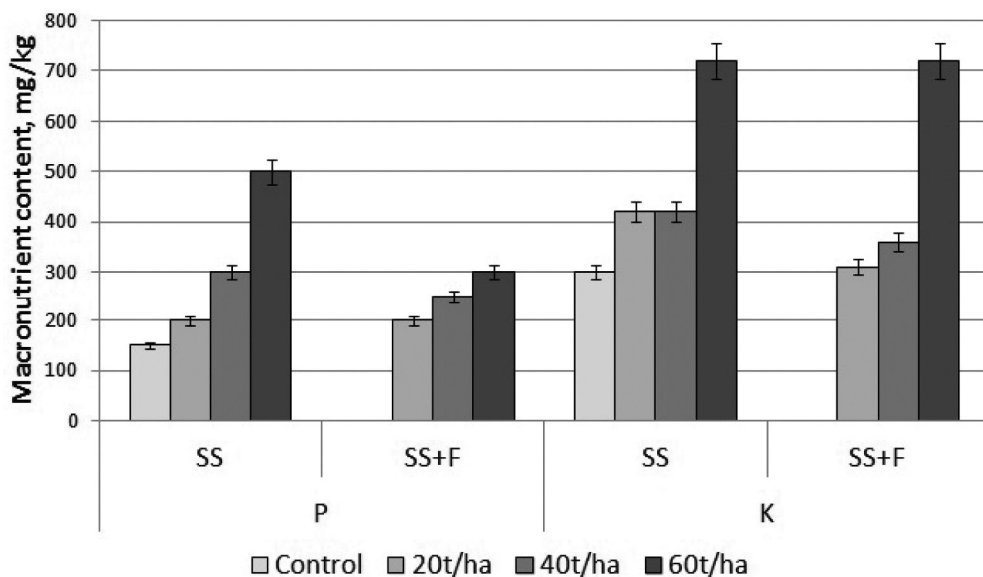


Figure 3. Content of phosphorus and potassium in miscanthus biomass.

The introduction of flocculated sewage sludge increased the nitrogen uptake 4.1–6.3 times (Figure 4). The uptake of potassium ranged from 2.4 kg/ha in the control to 8.2 and 9.3 kg/ha in variants receiving sludge at a dose of 60t/ha, an increase in the uptake of this element by 1.3–3.9 times, depending on the type and dose of sludge. The uptake of phosphorus was the smallest; it amounted to 1.2 kg/ha in the control. The introduction of

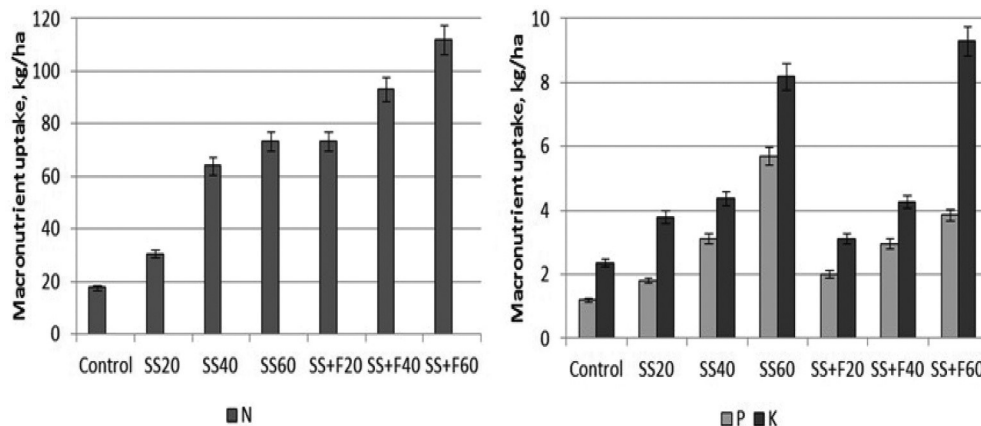


Figure 4. NPK uptake with biomass of 2-year-old miscanthus.

a small dose of SS increased this indicator by 1.5–1.8 times; at a dose of 40t/ha, the uptake increased by 2.5–2.6 times; and with SS at a dose of 60t/ha, uptake level increased by 3.2– 4.8 times. Whereas the uptake of nitrogen and potassium was increased to a greater extent by SS+F, the uptake of phosphorus responded more to the application of simple SS.

The content of available to plants of trace elements, including toxic ones in loess-like loam, was recorded in small amounts (Table 2).

The introduction of sewage sludge increased the content of iron and manganese by 43– 49%; the copper content increased by 6–26%; in all three cases with a more pronounced effect from the introduction of a simple sewage sludge. In contrast, the introduction of sewage sludge in rate of 20 and 40 t/ha led to an increase in the zinc content in the range of 6–46% (SS+F) and 37–64% (SS); the dose of 60 t/ha increasing the amount of zinc by 2.3– 2.9 times. The same dynamics were observed in the case of cobalt. The use of sewage sludge contributed to an increase in the content of nickel and lead in the substrate by 12–88%; nickel content was more in the SS+F trial and lead in the SS treatment. The introduction of a simple sewage sludge led to a significant increase in the content of chromium (3–9 times) and cadmium (3–5.5 times); whereas, using flocculated sludge, this increase was 2–3.4 times. At the same time, the content of chromium and cadmium was within the normal range, even with the introduction of large doses of sewage sludge.

Table 2. Available trace elements, average content in loess-like loam, mg/kg (N = 3).

Elements	SS				SS+F		
	Control	20t/ha	40t/ha	60t/ha	20t/ha	40t/ha	60t/ha
Fe	11.55 ± 0.6	12.8 ± 0.7	15.6 ± 0.7	16.2 ± 0.9	11.8 ± 0.6	14.2 ± 0.6	15.9 ± 0.7
Mn	35.3 ± 1.1	35.5 ± 1.2	37.5 ± 1.4	52.9 ± 1.7	37.8 ± 1.3	38.4 ± 1.3	51.8 ± 1.6
Cu	1.22 ± 0.09	1.44 ± 0.1	1.47 ± 0.12	1.54 ± 0.12	1.29 ± 0.1	1.36 ± 0.13	1.40 ± 0.11
Zn	2.9 ± 0.12	4.0 ± 0.30	4.78 ± 0.36	8.58 ± 0.43	3.1 ± 0.26	4.25 ± 0.35	6.9 ± 0.38
Co	0.25 ± 0.03	0.28 ± 0.04	0.34 ± 0.04	0.53 ± 0.05	0.29 ± 0.03	0.35 ± 0.03	0.45 ± 0.04
Ni	0.56 ± 0.06	0.63 ± 0.07	0.78 ± 0.07	0.91 ± 0.08	0.57 ± 0.06	0.82 ± 0.08	1.03 ± 0.09
Cr	0.1 ± 0.01	0.34 ± 0.02	0.76 ± 0.04	0.92 ± 0.05	0.12 ± 0.01	0.20 ± 0.02	0.31 ± 0.02
Pb	1.41 ± 0.09	1.65 ± 0.12	1.95 ± 0.14	2.66 ± 0.16	1.66 ± 0.10	1.99 ± 0.11	2.19 ± 0.15
Cd	0.05 ± 0.01	0.17 ± 0.02	0.25 ± 0.04	0.28 ± 0.04	0.11 ± 0.02	0.13 ± 0.02	0.17 ± 0.02

The increase in trace element concentration on application of sludge should be taken into account if it is used on soils contaminated with these metals. Among the minor essential elements, the highest content in the miscanthus biomass was for iron and manganese. Both increased under the application of sludge, according to the rate of application (Figure 5). Sewage sludge had a greater effect on iron content than SS+F, whereas the effect of both types of sludge on manganese was much the same.

The content of zinc and, especially, copper in the above-ground biomass was significantly lower than iron and manganese (Figure 6) but sludge application led to an increase in the content of both elements. Zinc increased by 2.8 and 3.2 times in the SS +F60 and SS60 treatments, respectively. The application of SS at doses of 40 and 60t/ha

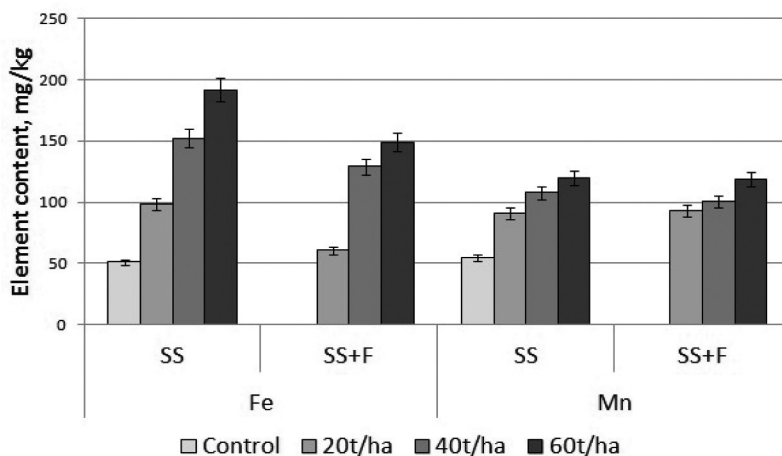
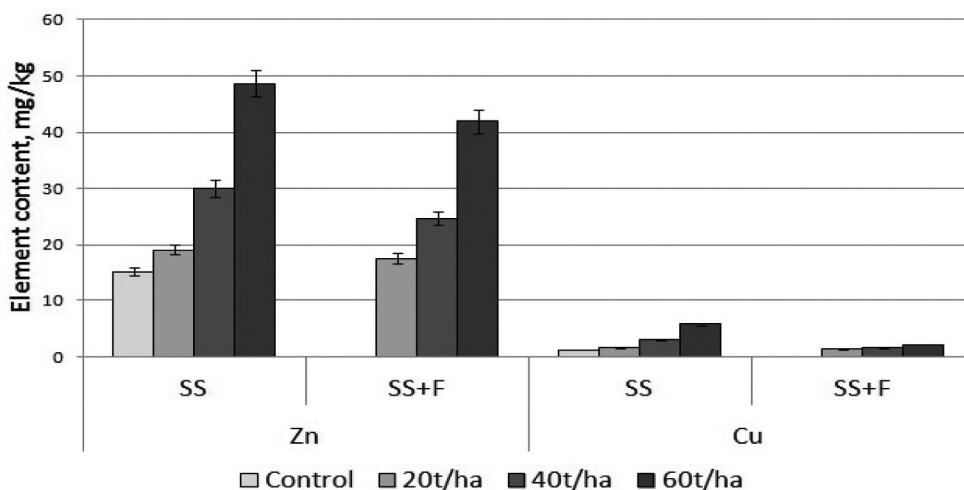


Figure 5. Content of iron and manganese in miscanthus above-ground biomass.



6. Content of zinc and copper in miscanthus above-ground biomass.

Figure

had a significant effect on copper, which increased by 2.4–3.7 times. The increase was only 1.2–1.8 times with the same doses of SS+F.

The intensity of accumulation of minor essential elements can be arranged in order: Fe (mBCF = 5.1–11.8) → Zn (mBCF = 4.7–6.3) → Mn (mBCF = 1.5–2.8) → Cu (mBCF = 1.0–2.1).

Depending on the type of sewage sludge and application dose, the uptake of iron per hectare varied within 0.4–1.9 kg, manganese 0.4–1.5 kg, zinc 0.1–0.55 kg. Uptake of copper was very small, within 10 g/ha (control) to 70/ha (SS60) (Figures 7 and 8).

Among the heavy metals, the highest values in the above-ground biomass were noted for lead and chromium (Figure 9). The application of sewage sludge contributed to an increase in the content of heavy metals in biomass by 1.1–2.3 times for cobalt, nickel and cadmium, by 1.1–1.8 times for chromium, and by 1.2–2.6 times for lead. Chromium is

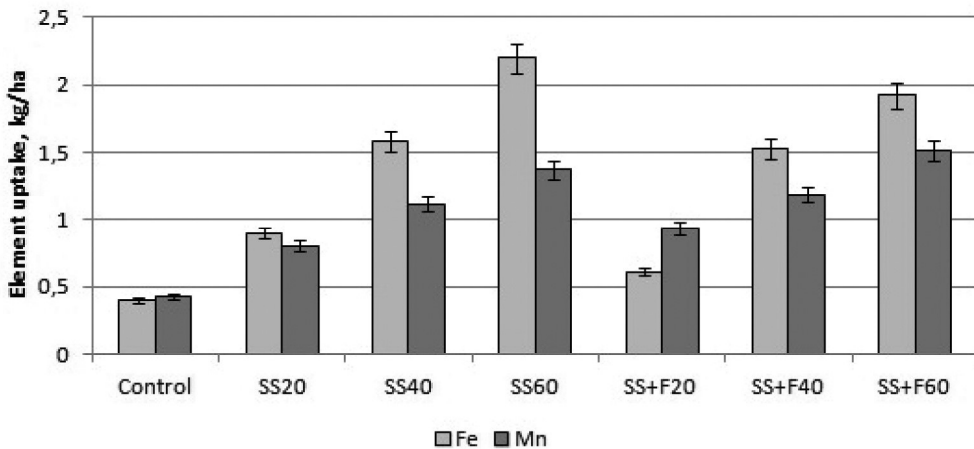
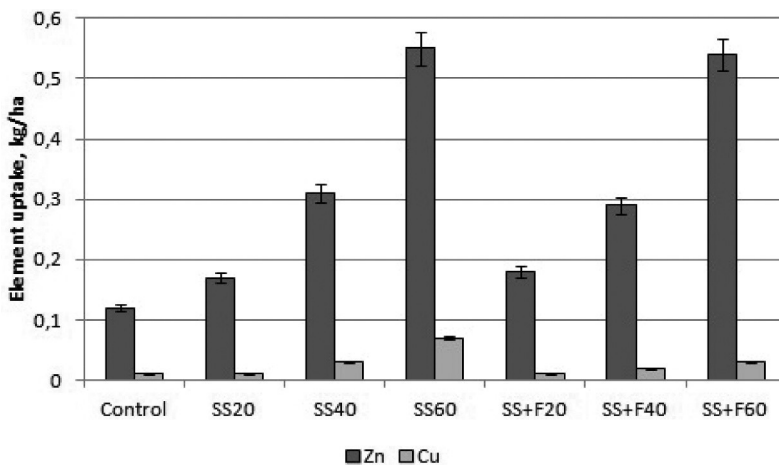


Figure 7. Uptake of iron and manganese by above-ground biomass of 2-year-old miscanthus.



Figure

8. Uptake of zinc and copper in miscanthus above ground biomass.

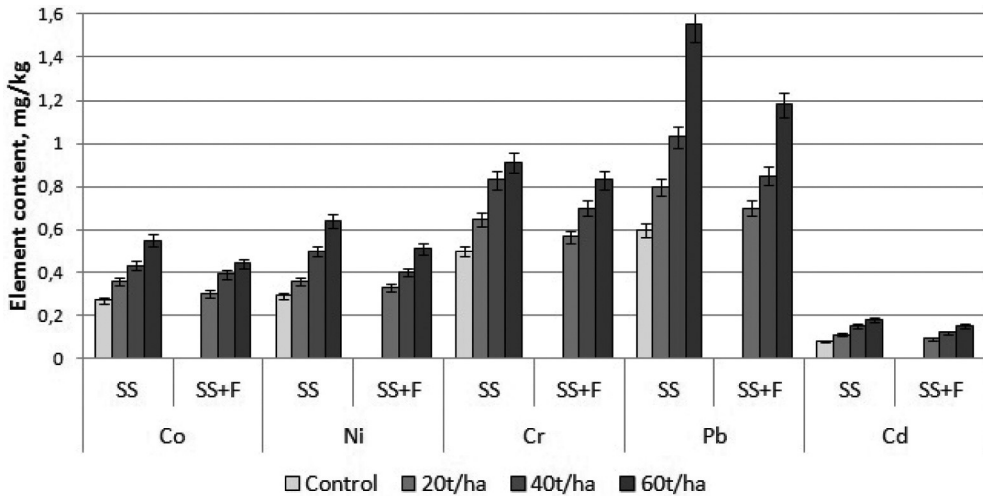
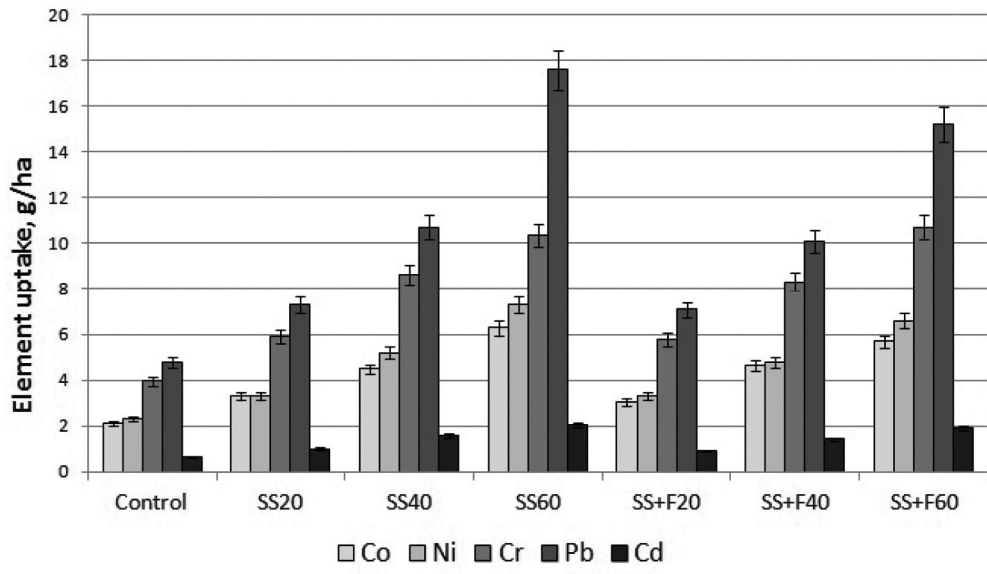


Figure 9. Content of heavy metals in above-ground miscanthus biomass.

actively absorbed; its mBCF values varied from 2.0 to 4.9, depending on the treatment, whereas for lead, cobalt, nickel and cadmium they did not exceed 0.5–0.9. Simple sewage sludge had a greater effect than flocculated sludge.

The uptake of heavy metals by the miscanthus biomass over two years is small (Figure 10): the highest values were for lead (15.2–17.6 g/ha) and chromium (10.4– 10.7 g/ha) in the SS60 and SS+F60 trials. The uptake of cadmium was the smallest and did not exceed 0.6–2.0 g/ha. The uptake of chromium increased by 1.4–2.6 times, the



10. Heavy metal uptake by above-ground biomass of 2-year-old miscanthus.

uptake of cadmium and nickel by 1.4–3.2 times, cobalt by 1.5–6.3 times, depending on the type of SS and the dose of application.

### *Thermogravimetric analysis*

Thermal decomposition of miscanthus biomass proceeds in the temperature range of 40–580°C in four stages (Table 3). The first stage of evaporation of water and decomposition of volatile components takes place at relatively low temperatures of 45–150°C with a weight loss of 11.1%. One degradation peak at 80°C with a maximum weight loss rate of 6.5%/min was observed in this range.

Decomposition of the main components of biomass (hemicellulose, cellulose and lignin) occurs in the second to fourth stages. Decomposition of hemicellulose took place at 170–280°C. The rate of decomposition is much higher than at the previous stage, peak degradation was observed at a temperature of 260°C, and the weight loss was 14.9%. In the phase of cellulose decomposition (280–390°C), peak destruction was at 320°C, the maximum rate was 28.2%/min, and the weight loss at this stage was also the largest – 38.3%. Cleavage of lignin took place during the last two periods, relatively slowly without pronounced peaks. The formation of an incombustible residue occurs at the last stage; an inconspicuous peak was observed at 440°C. A total of 33.9% of the mass was lost at this stage. The proportion of non-combustible residue was 1.8%.

The introduction of sewage sludge changes the thermal characteristics of the biomass. In the experiment with SS, the duration of thermolysis is reduced, the rates of process

**Table 3.** Main parameters of miscanthus biomass thermal destruction.

Trial	Stage	Interval, °C	Peak, °C	Maximum rate, %/min	Mass loss, %	Share of residual mass, %
Control	I	40–150	80	6.5	11.1	
	II	150–280	260	18.5	14.9	
	III	280–390	320	28.2	38.3	
	IV	390–580	440	8.2	33.9	1.8
SS20	I	70–180	130	6.4	10.0	
	II	180–270	240	24.4	27.3	
	III	270–380	300	33.4	37.7	
	IV	380–540	420	7.6	24.1	0.9
SS40	I	60–170	130	6.4	10.1	
	II	170–270	270	17.9	27.3	

	III	270–380	310	25.1	36.1	
	IV	380–520	425	7.7	25.0	1.2
SS60	I	60–180	130	5.8	10.0	
	II	180–260	255	19.2	22.8	
	III	260–390	300	28.9	38.6	
	IV	390–520	420	8.0	27.7	0.9
SS+F20	I	70–180	100	5.8	9.0	
	II	180–260	220	17.1	29.0	
	III	260–380	300	25.6	35.9	
	IV	380–500	420	7.2	25.1	1.0
SS+F40	I	50–180	130	5.1	6.6	
	II	180–270	240	15.8	26.7	
	III	270–380	300	23.5	39.2	
	IV	380–550	425	6.9	24.5	3.0
SS+F60	I	60–160	100	4.4	10.2	
	II	160–270	260	14.3	29.1	
	III	270–390	300	22.1	35.4	
	IV	390–520	420	6.5	21.5	3.8

change, and the degradation peaks shift; in all experimental variants, the decomposition peak of volatile components (the first stage) was shifted to higher temperatures. At the same time, the rate of processes did not differ much from the control values (Figure 11). In the SS20 and SS60 treatments, the rate of decomposition of hemicellulose and cellulose was significantly higher than in the control; the degradation peaks shifted to lower temperatures. The second and third stages in the SS40 treatment were the closest to the control indicators.

The thermal effects in the test samples were more pronounced in the temperature range of 300–420°C than in the control (Figure 12). The introduction of SS contributed to a more complete combustion of biomass; the share of residual mass decreased by 33–50%.

Flocculated sludge also affected the thermal performance of the biomass. At the first stage, the destruction peaks again shifted to higher temperatures (Figure 13). At the stage of hemicellulose decomposition, the peak shift to lower temperatures

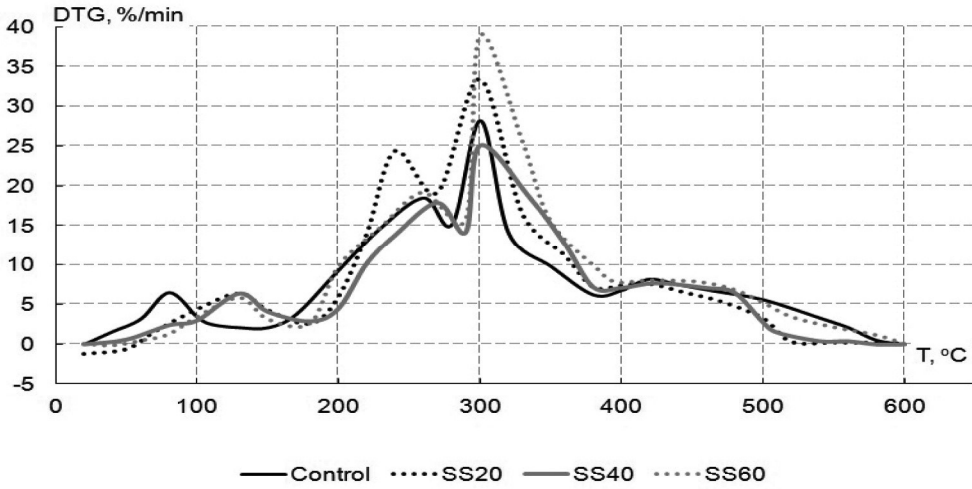


Figure 11. DTG curves of miscanthus biomass thermal destruction.

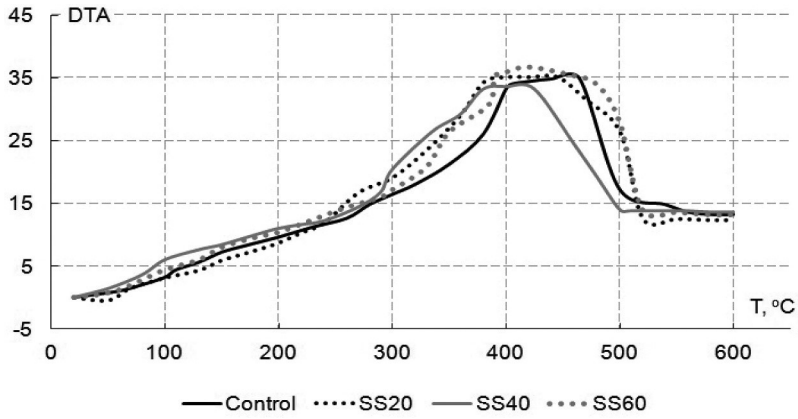
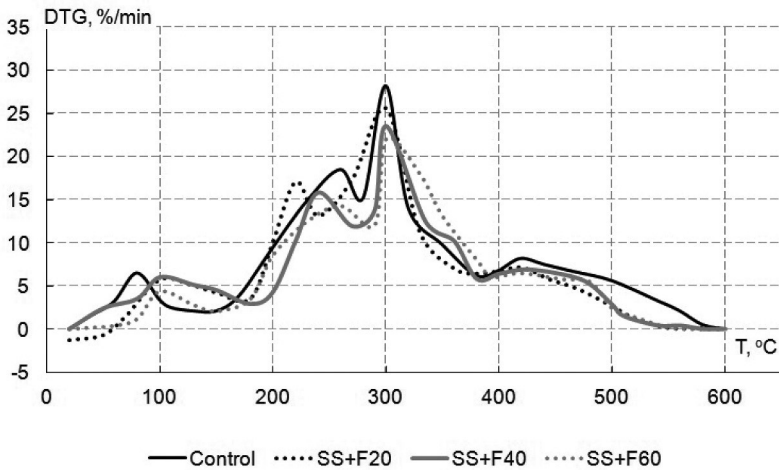


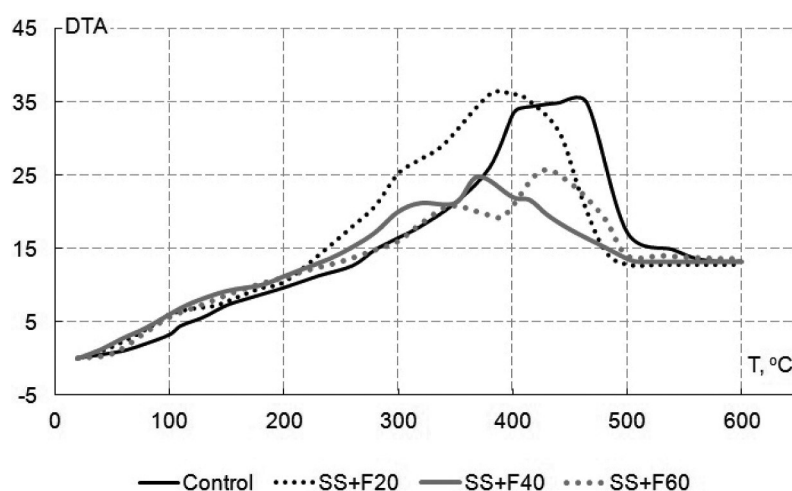
Figure 12. Thermal effects of miscanthus biomass thermolysis.



**Figure 13.** DTG curves of miscanthus biomass thermal destruction.

was noted only in the SS+F20 trial. The decomposition of cellulose in the test samples was similar to the control, but the rates were somewhat lower. There were no special differences between the experimental and control samples at the last stage of thermal destruction. Biomass combustion was more complete only in the SS+F20 trial.

The thermal effects of exothermic reactions of the SS+F20 trial in the temperature range of 220–400°C were more pronounced than in the control and other experimental samples (Figure 14). In the range from 420°C to 500°C, thermal effects in all experimental samples were less noticeable.



**Figure 14.** Thermal effects of miscanthus biomass thermolysis.

## Discussion

At present, only 25% of sewage is treated to a level that meets current hygiene standards [48,49].

There are various flocculants for removing suspended and dissolved solids, colloids and organic matter from the wastewater [50–52]. The content of total carbon and total nitrogen in the sewage sludge treated with DAMET polymer increased by 1.5 times. At the same time, the share of total phosphorus decreased by half, but the amount of total potassium remained at the same level. The mass fraction of manganese decreased to one-third, copper to half, zinc more than 1.5 times. The levels of cobalt, nickel, lead, chromium and cadmium in the two samples are very low.

The introduction of sewage sludge increased miscanthus biomass productivity in proportion to the rates of application (from 20 to 60 tonnes/ha), comparable to similar field experiments in Poland on clay loam soil where rates of 10, 20, 40 and 60 tonnes/ha were applied over 6 years [27]. The Polish experiments indicated that the proportional

effect of SS was greatest at the 20 tonnes/ha and the highest heavy metals (Cr, Ni, Pb, Cd and Zn) uptake was also under the 20t/ha dose, excluding Cu. These and other similar researches show the adaptive potential of miscanthus [53–55]. In our case, the heavy metals uptake by miscanthus increased in proportion to the application of sludge. It appears that the buffer capacity of our loess-like loam was inferior to the absorption capacity of these elements by clay loam in the experiment of our Polish colleagues. It was also demonstrated that miscanthus growth depended more on the silt content of the soil rather than trace elements in the sludge [56].

Intensive accumulation of heavy metals in the biomass of energy plants fertilised by sewage sludge soil can create new environmental issues [57]. The prospect of decreasing heavy metal uptake from contaminated soil was shown in a pot experiment using 3% biochar [58], but the ash from combustion of miscanthus pellets might nevertheless qualify as environmentally hazardous waste [26]. That is why we have paid attention to the share of incombustible residue after combustion of miscanthus biomass taken in the trials.

## Conclusions

- The introduction of sewage sludge increases the yield of biomass and content of nitrogen, phosphorus and potassium in the soil and in crops. The application of flocculated sludge (SS+F) had a greater effect on the accumulation of nitrogen compared to the simple sewage sludge (SS); nitrogen uptake increased by 4.1–6.3 times. In contrast, sludge had a greater effect than flocculated sludge on the accumulation of phosphorus and potassium.
- The maximum uptake of minor essential elements by the miscanthus biomass over 2 years was 1.9 kg/ha for iron, 1.5 kg/ha for manganese, 0.55 kg/ha for zinc, and 0.07 kg/ha for copper. The intensity of accumulation of minor essential elements can be arranged in order: Fe (mBCF = 5.1–11.8) → Zn (mBCF = 4.7–6.3) → Mn (mBCF = 1.5–2.8) → Cu (mBCF = 1.0–2.1).
- The modified bioconcentration factors for lead, cobalt, nickel and cadmium do not exceed 0.5–0.9, whereas chromium is actively absorbed (mBCF = 2–4.9). The uptake of heavy metals by miscanthus biomass over 2 years was small: the highest values were of lead (15.2–17.6 g/ha) and chromium (10.4–10.7 g/ha) in the SS60 and SS60 +F trials. The uptake of cadmium was the smallest and did not exceed 0.6–2.0 g/ha.
- The introduction of sewage sludge affects the thermal characteristics of miscanthus biomass; the effect of SS is more pronounced than the effect of SS+F. There is a reduction in the duration of the thermolysis, an increase or decrease in the rates of processes, a shift of degradation peaks towards lower or higher temperature areas. In addition, all SS variants and SS+F20 contribute to a more complete combustion of biomass, whereas SS+F at doses of 40 and 60t/ha increases the share of incombustible residue.

## Disclosure statement

No potential conflict of interest was reported by the author(s).

## ORCID

Mykola Kharytonov  <http://orcid.org/0000-0002-4650-5819>

Liliya Frolova  <http://orcid.org/0000-0001-7970-2264>

## References

- [1] Volungevičius, J., Feiza, V. Amalevičiūtė-Volungė, K., et al., 2019, Transformations of different soils under natural and anthropogenized land management. *Zemdirbyste-Agriculture* **106**(1), 3–14. doi:10.13080/z-a.2019.106.001.
- [2] Velichka, R., Rimkevichene, M. and Martsinkyavichene, A., 2006, Changes in the properties of a loamy Gleyic Cambisol as related to the saturation of crop rotations with rape. *Eurasian Soil Science* **39**(9), 1002–1010. doi:10.1134/S1064229306090109.
- [3] Macia, P., Fernandez-Costas, C. Rodrigues, E., et al., 2014, Technosols as novel valorization strategy for an ecological management of dredged marine sediments. *Ecological Engineering* **67**, 182–189. doi:10.1016/j.ecoleng.2014.03.020.
- [4] Kharytonov, M., Pidlisnyuk, V. Stefanovska, T., et al., 2019, The estimation of miscanthus × giganteus adaptive potential for cultivation on the mining and post-mining lands in Ukraine. *Environmental Science Pollution Research International* **26**(3), 2974–2986. doi: 10.1007/s11356-018-3741-0.
- [5] Betancur-Corredor, B., Loaiza-Usuga, J.C. Denich, M., et al., 2020, Changes of technosol properties and vegetation structure along a chronosequence of dredged sediment deposition in areas with alluvial gold mining in Colombia. *Journal of Soils & Sediments* **20**, 2377–2394. doi:10.1007/s11368-019-02551-9.
- [6] Tarika, O. and Zabaluev, V., 2000, Mine land reclamation strategies in the Nikopol manganese ore basin (Central steppe of Ukraine): Using replaced mining overburden in agriculture. In: *Proceedings of the 16th annual conference of the society for ecological restoration*, Victoria, BC, Canada.
- [7] Séré, G., Schwartz, C. Ouvrard, S., et al., 2008, Soil construction: A step for ecological reclamation of derelict lands. *Journal of Soils Sediments* **8**, 130–136. doi:10.1065/jss2008.03.277.
- [8] Aggelides, S.M. and Londra, P.A., 2000, Effects of compost produced from town wastes and sewage sludge on the physical properties of a loamy and a clay soil. *Bioresource Technology* **71**(3), 253–259. doi:10.1016/S0960-8524(99)00074-7.
- [9] Liu, J., Schulz, H. Brandl, S., et al., 2012, Short-term effect of biochar and compost on soil fertility and water status of a Dystric Cambisol in NE Germany under field conditions. *Journal of Plant Nutrition and Soil Science* **175**, 698–707. <https://api.semanticscholar.org/CorpusID:95450741>
- [10] Bielski, S., 2015, The agricultural production of biomass for energy purposes in Poland. *Agriculture and Forestry* **61**(1), 153–160. doi:10.17707/agricultforest.61.1.20.
- [11] Blanco-Canqui, H., 2016, Growing dedicated energy crops on marginal lands and ecosystem services. *Soil Science Society of America Journal* **80**(4), 845–858. doi:10.2136/sssaj2016.03.0080.
- [12] Kharytonov, M., Babenko, M. Martynova, et al., 2023, Elemental composition of miscanthus biomass grown on different kinds of Technosols. *The International Journal of Environmental Studies* **80**(2), 488–496. doi:10.1080/00207233.2022.2152624.

- [13] Tsapko, Y., Starshenko, O. and Vodiak, Y., 2023, Using the ecosystem services potential of chernozem to restore war-damaged land. *International Journal of Environmental Studies* **80** (2), 399–409. doi:10.1080/00207233.2023.2179760.
- [14] Lopushnyak, V., Hrytsulyak, H. Lopushniak, H., et al., 2023, Integrated assessment of pollution of sod-podzolic soils by application of sewage sludge under *Miscanthus x giganteus*. *The International Journal of Environmental Studies* **80**(2), 476–487. doi:10.1080/00207233.2022.2147725.
- [15] Heaton, E.A., Dohleman, F.G. and Long, S.P., 2008, Meeting US biofuel goals with less land: The potential of miscanthus. *Global Change Biology* **14**, 2000–2014. doi:10.1111/j.1365-2486.2008.01662.x.
- [16] Wanat, N., Austruy, A. Jousen, E., et al., 2013, Potential of miscanthus x giganteus grown on highly contaminated Technosols. *Journal of Geochemical Exploration* **126–127**, 78–84. doi:10.1016/j.gexplo.2013.01.001.
- [17] Haines, S.A., Gehl, R.J., Havlin, J.L. and Ranney, T.G., 2015, Nitrogen and phosphorus fertilizer effects on establishment of giant miscanthus. *BioEnergy Research* **8**(1), 17–27. doi:10.1007/s12155-014-9499-4.
- [18] Winkler, B., Mangold, A. von Cossel, M., et al., 2020, Implementing miscanthus into farming systems: A review of agronomic practices, capital and labour demand. *Renewable and Sustainable Energy Reviews* **132**, 110053. doi:10.1016/j.rser.2020.110053.
- [19] Werle, S., Bisorca, D. Katelbach-Wozniak, A., et al., 2016, Phytoremediation as an effective method to remove heavy metals from contaminated area - TG/FT-IR analysis results of the gasification of heavy metal contaminated energy crops. *Journal of the Energy Institute* **90**, 408–417. doi:10.1016/j.joei.2016.04.002.
- [20] Kocoń, A. and Jurga, B., 2017, The evaluation of growth and phytoextraction potential of miscanthus x giganteus and sida hermaphrodita on soil contaminated simultaneously with Cd, Cu, Ni, Pb, and Zn. *Environmental Science and Pollution Research* **24**(5), 4990–5000. doi:10.1007/s11356-016-8241-5.
- [21] Baker, A.J.M., Reeves, R.D. and Hajar, A.S.M., 1994, Heavy metal accumulation and tolerance in British populations of the metallophyte. *The New Phytologist* **127**(1), 61–68. doi:10.1111/j.1469-8137.1994.tb04259.x.
- [22] Barbosa, B., Boléo, S. Sidella, S., et al., 2015, Phytoremediation of heavy metal-contaminated soils using the perennial energy crops miscanthus spp. And *Arundo donax* L. *BioEnergy Research* **8**, 1500–1511. doi:10.1007/s12155-015-9688-9.
- [23] Pidlisnyuk, V., Aigerim Mamirova, A., Kumar Pranaw, K., Stadnik, V., Kuráň, P., Trögl, J. Shapoval, P., et al., 2022, Miscanthus x giganteus phytoremediation of soil contaminated with trace elements as influenced by the presence of plant growth-promoting bacteria. *Agronomy* **12**(4), 771. doi:10.3390/agronomy12040771.
- [24] von Cossel, M., Mangold, A. Iqbal, Y., et al., 2019, How to generate yield in the first year – a three-year experiment on miscanthus (*miscanthus x giganteus* Greef et Deuter). Establishment under maize (*Zea mays* L.). *Agronomy* **9**, 237. doi:10.3390/agronomy9050237.
- [25] Kołodziej, B., Antonkiewicz, J. and Sugier, D., 2016, Miscanthus giganteus as a biomass feedstock grown on municipal sewage sludge. *Industrial Crops and Products* **81**, 72–82. doi:10.1016/j.indcrop.2015.11.052.
- [26] Antonkiewicz, J., Kołodziej, B. and Bielińska, E.J., 2016, The use of reed canary grass and giant miscanthus in the phytoremediation of municipal sewage sludge. *Environmental Science and Pollution Research* **23**(10), 9505–9517. doi:10.1007/s11356-016-6175-6.
- [27] Antonkiewicz, J., Kołodziej, B., Bielińska, E.J. and Popławska, A., 2019, The possibility of using sewage sludge for energy crop cultivation exemplified by reed canary grass and giant miscanthus. *Soil Science Annual* **70**(1), 21–33. doi:10.2478/ssa-2019-0003.
- [28] Ociepa-Kubicka, A. and Pachura, P., 2013, The use of sewage sludge and compost for fertilization of energy crops on the example of miscanthus and Virginia mallow. *Rocznik Ochrona Środowiska* **15**, 2267–2278.

- [29] Dubis, B., Szatkowski, A. and Jankowski, K.J., 2022, Sewage sludge, digestate, and mineral fertilizer application affects the yield and energy balance of Amur silvergrass. *Industrial Crops and Products* **175**, 114235. doi:10.1016/j.indcrop.2021.114235.
- [30] Dubis, B., Jankowski, K., Załuski, D. and Sokolski, M., 2020, The effect of sewage sludge fertilization on the biomass yield of giant miscanthus and the energy balance of the production process. *Energy* **206**, 118189. doi:10.1016/j.energy.2020.118189.
- [31] Matusiak, M., Ślęzak, R. and Ledakowicz, S., 2020, Thermogravimetric kinetics of selected energy crops pyrolysis. *Energies* **13**(15), 3977. doi:10.3390/en13153977.
- [32] Mészáros, E., Jakab, E., Várhegyi, G. and Tovar, P., 2007, Thermogravimetry/mass spectrometry analysis of energy crops. *Journal of Thermal Analysis and Calorimetry* **88**, 477–482. doi:10.1007/s10973-006-8102-4.
- [33] Arundale, R.A., Bauer, S. Haffner, F.B., et al., 2015, Environment has little effect on biomass biochemical composition of *miscanthus* × *giganteus* across soil types, nitrogen fertilisation, and times of harvest. *BioEnergy Research* **8**, 1636–1646. doi:10.1007/s12155-015-9613-2.
- [34] Peni, D., Stolarski, M.J. and Dębowski, M., 2022, Green biomass quality of perennial herbaceous crops depending on the species, type and level of fertilization. *Industrial Crops and Products* **184**, 115026. doi:10.1016/j.indcrop.2022.115026.
- [35] Ozdemir, S., Dede, O.H., Inan, M. and Turp, S.M., 2018, Effects of sewage sludge on energy content and combustion emissions of energy crops. *International Journal of Agriculture and Biology* **20**, 1575–1580. doi:10.17957/IJAB/15.0671.
- [36] Havryushenko, O., Mytsyk, O. Kharytonov, M., et al., 2022, The suitability of physical and chemical properties of rocks for land reclamation in different subzones of the Ukrainian steppe. *Journal of Geology, Geography and Geoecology* **31**(2), 251–259. doi:10.15421/112223.
- [37] Kharytonov, M., Martynova, N. Babenko, et al., 2023, Production of sweet sorghum bio-feedstock on technosol using municipal sewage sludge treated with flocculant in Ukraine. *Agriculture* **13**, 1129. doi:10.3390/agriculture13061129.
- [38] Nelson, D.W. and Sommers, L.E., 1996, Total carbon, organic carbon and organic matter. In: J.M. Bigham (Ed.) *Methods of soil analysis, part 3 chemical methods*. SSSA book series 5. (Madison, WI: Soil Science Society of America and American Society of Agronomy), pp. 1001–1006.
- [39] Cornfield, A.H., 1960, Ammonia released on treating soils with N sodium hydroxide as a possible method for predicting the nitrogen supplying power of soils. *Nature (London)* **187**, 260–261. doi:10.1038/187260a0.
- [40] Olsen, S.R., Cole, C.V. Watanabe, F.S., et al., 1954, *Estimation of available phosphorus in soils by extraction with sodium bicarbonate*. USDA circular 939 (Washington, DC: USDA).
- [41] Biswanath, D., Rogers, C.W. Leytem, A.B., et al., 2019, Evaluation of soil test phosphorus extractants in Idaho soils. *Soil Science Society of America Journal* **83**(3), 817–824. doi:10.2136/sssaj2018.08.0314.
- [42] Khan, M.I., Afzal, M.J. Bashir, S., et al., 2021, Improving nutrient uptake, growth, yield and protein content in chickpea by the co-addition of phosphorus fertilizers, organic manures, and bacillus sp. MN-54. *Agronomy* **11**, 436. doi:10.3390/agronomy11030436.
- [43] Kabata-Pendias, A., 2004, Soil-plant transfer of trace elements - An environmental issue. *Geoderma* **122**(2–4), 143–149. doi:10.1016/j.geoderma.2004.01.004.
- [44] Haigh, M. and Dyckhoff, C., 1996, Soils. In: S. Watts and L. Halliwell (Eds) *Essential environmental science, methods & techniques* (London: Routledge), pp. 261–303.
- [45] Thomas, R.L., Sheard, R.W. and Mayer, J.R., 1967, Comparison of conventional and automated procedures for nitrogen, phosphorus and potassium analysis of plant material using a single digestion. *Agronomy Journal* **59**, 240–243. doi:10.2134/agronj1967.00021962005900030010x.
- [46] Przygocka-Cyna, K. and Grzebisz, W., 2018, Effect of biofertilizers on nutrient uptake by vegetables grown in a short cropping sequence. *Journal of Elementology* **23**(3), 807–823. doi:10.5601/jelem.2017.22.4.1479.

- [47] Paulik, F., Paulik, J. and Erdey, L., 1966, Derivatography. A complex method of thermal analysis. *Talanta* **13**(10), 1405. doi:[10.1016/0039-9140\(66\)80083-8](https://doi.org/10.1016/0039-9140(66)80083-8).
- [48] Svirenko, L. and Bondar, V., 2010, Sludge treatment for application in agriculture should be the option number one for Ukraine. *Linnaeus ECO-TECH* **10**, 356–362. doi:[10.15626/Eco-Tech.2010.037](https://doi.org/10.15626/Eco-Tech.2010.037).
- [49] Vankovych, D., Bota, O. Malovanyy, M., et al., 2021, Assessment of the prospects of application of sewage sludge from Lviv wastewater treatment plants for the purpose of conducting the biological reclamation. *Journal of Ecological Engineering* **22**(2), 134–143. doi:[10.12911/22998993/130892](https://doi.org/10.12911/22998993/130892).
- [50] Lee, C.S., Robinson, J. and Chong, M.F., 2014, A review on application of flocculants in wastewater treatment. *Process Safety and Environmental Protection* **92**(6), 489–508. doi:[10.1016/j.psep.2014.04.010](https://doi.org/10.1016/j.psep.2014.04.010).
- [51] Renault, F., Sancey, B. Charles, J., et al., 2009, Chitosan flocculation of cardboard-mill secondary biological wastewater. *Chemical Engineering Journal* **155**(3), 775–783. doi:[10.1016/j.cej.2009.09.023](https://doi.org/10.1016/j.cej.2009.09.023).
- [52] Yin, X., Li, Z., Chai, F., Tian, Y., Tan, Y. and Wang, M., et al., 2021, Synthesis of a micro-crosslinked polyacrylamide flocculant and its application in treatment of oily produced water. *Energy & Fuels* **35**(22), 18396–18405. doi:[10.1021/acs.energyfuels.1c02213](https://doi.org/10.1021/acs.energyfuels.1c02213).
- [53] Borkowska, H. and Molas, R., 2013, Yield comparison of four lignocellulosic perennial energy crop species. *Biomass and Bioenergy* **51**, 145–153. doi:[10.1016/j.biombioe.2013.01.017](https://doi.org/10.1016/j.biombioe.2013.01.017).
- [54] Li, C., Xiao, B. Wang, Q.H., et al., 2014, Phytoremediation of Zn and Cr-contaminated soil using two promising energy grasses. *Water, Air, and Soil Pollution* **225**, 2027. doi:[10.1007/s11270-014-2027-5](https://doi.org/10.1007/s11270-014-2027-5).
- [55] Pavel, P.B., Puschenreiter, M. Wenzel, W.W., et al., 2014, Aided phytostabilization using miscanthus sinensis × giganteus on heavy metal-contaminated soils. *Science of the Total Environment* **479–480**, 125–131. doi:[10.1016/j.scitotenv.2014.01.097](https://doi.org/10.1016/j.scitotenv.2014.01.097).
- [56] Nsanganwimana, F., Al Souki, K.S. Waterlot, C., et al., 2021, Potentials of miscanthus x giganteus for phytostabilization of trace element-contaminated soils: Ex situ experiment. *Ecotoxicology & Environmental Safety* **214**, 112125. doi:[10.1016/j.ecoenv.2021.112125](https://doi.org/10.1016/j.ecoenv.2021.112125).
- [57] Pidlisnyuk, V., Stefanovska, T. Lewis, E.E., et al., 2014, Miscanthus as a productive biofuel crop for phytoremediation. *Critical Reviews in Plant Science* **33**(1), 1–19. doi:[10.1080/07352689.2014.847616](https://doi.org/10.1080/07352689.2014.847616).
- [58] Kharytonov, M., Klimkina, I., Martynova, N., et al., 2019, The biochar impact on miscanthus and sunflower growth in marginal lands. *Agrology* **3**(1), 3–11. doi:[10.32819/020001](https://doi.org/10.32819/020001).