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Full Length Article

Effects of Sewage Sludge on Energy Content and Combustion Emissions of Energy Crops

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Abstract

This study was conducted to investigate the energy crop production suitability of sewage sludge as well as pollutant transfer from sewage sludge to feedstock of *Miscanthus x giganteus* and *Arundo donax*, with a focus on ash constituents and exhaust gaseous emissions. Dewatered sludge and natural field soil were filled into pots, and seedlings produced from rhizomes were planted in the pots, which were arranged under a completely randomized design with four replications. The soil treatments were fertilized with conventional fertilizer, while no external fertilizer was supplied for sludge treatments. Harvested plant samples were subjected to characterization. Both crop species produced sufficient biomass yield on either soil or sludge treatments. High heating values measured for plants cultivated on soil and sludge were insignificant and in the range of 17.95–17.62 MJ kg^{-1} and 17.31–18.05 MJ kg^{-1} for *M. x giganteus* and *A. donax*, respectively. Sewage sludge significantly increased the plant macronutrients and heavy metal content of both species. Si was the dominant element in ash and was higher in *A. donax* plants. K and Ca were secondary high concentration elements, giving similar results for both plant species. Nitrogen concentrations in plant tissue provided by sewage sludge increased NO_x emissions with a maximum increment observed at 10% with *A. donax* plants. SO_2 emissions obtained were consistent with plant species but the emission concentrations were very small. The use of sewage sludge for energy crop cultivation confirmed that ash and air emissions hold great potential for both the energy crop production and waste minimizing alternative.

Keywords: *Arundo donax*; Emissions; Energy; *Miscanthus x giganteus*; Sewage sludge

Introduction

Energy plant cultivation is receiving great attention due to the progressing depletion of fossil fuels and as a sustainable resource, is considered to be a primary energy source especially in energy-poor countries such as Turkey (Balat, 2005). Among energy crops, fast growing and high dry biomass producing trees and perennial grass species present great potential to produce biomass feedstock (Baxter *et al*., 2014). These woody and grass species contain mainly lignin, cellulose and hemicellulose, which is rich in calorific values. Regarding biomass yield, grass species produce twice as much dry mass than woody species. For example, Angelini *et al*. (2009), reported that 30 t dry matter ha^{-1} yr^{-1} were obtained from giant miscanthus (*Miscanthus* x *giganteus*) and giant reed (*Arundo donax*) in an experimental plantation, which contained a high proportion of energy-rich compounds (Lygin *et al*., 2011).

Giant miscanthus and giant reed deserve special attention owing to their substantial potential growth rate, high biomass yield, ease of cultivation and response to fertilization or waste amendments (Smith and Slater, 2010; Kolodziej *et al*., 2016). Moreover, energy generated from those plants is much greater than energy expenditures in the production of biomass (Acaroglu and Aksoy, 2005; Angelini *et al*., 2009). The majority of studies that investigated the response of energy crops to fertilizer applications have used inorganic nutrient sources. For more sustainable production and long-term nutrients, the provision of organic waste resources is specifically important for high biomass yielding perennial energy plants that remove plenty of nutrients from the soil at each harvest cycle. Although sewage sludge has been scarcely studied as a natural fertilizer source for energy crops, the available data on biomass yields are promising (Ociepa-Kubicka *et al*., 2016). In addition to sustainable energy production, the use of sewage sludge for energy crop production allows sustainable sewage sludge disposal alternatives, saves mineral fertilizer, lowers nitrogen leaching from soil (Esperschuetz *et al*., 2016) and causes acidification of alkaline soil, which makes the nutrients more bioavailable to plants (Dede *et al*., 2015).

Traditionally, energy crop biomass is used in combustion for energy-related applications, mainly in utility boilers. Because sewage sludge may contain a high proportion of macronutrients and pollutants, combustion is

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considered an environmental risk due to heavy metals in biomass ash and gaseous emissions such as NO_x and SO_2. Hence, an understanding of the pollutant transfer from sludge to biomass feedstock, along with ash and gaseous emissions, is basic for enhanced waste recycling in energy crop cultivation.

Regarding the environmental concern, the use of sewage sludge in the production of different species of energy crops has been evaluated at different experimental conditions. Most of those studies accentuated the biomass productivity, bioavailability of macro plant nutrients, such as N, P and K, and concentrations of heavy metals in plant tissues and soil. Besides plant nutrients, undesired metals (e.g., Cd and Pb) are also acquired by plants that are present in sewage sludge or soil background. For instance, Lag-Brotons *et al*. (2014), investigated the sewage sludge compost application to *Cynara cardunculus* plants and found a 68% increase in biomass production and 40% increase in seed yield compared with the control treatment. Ociepa-Kubicka *et al*. (2016) indicated that application of sludge to *M. x giganteus* increased the plant tissue concentration of heavy metals as compared with conventional fertilization. Helios *et al*. (2014) cultivated the energy crop *Spartina pectinate* in a sewage sludge experiment and found that the sludge significantly improved feedstock yield, and combustion emissions were not deteriorated by waste residues. Likewise, Jeguirim *et al*. (2010) cultivated *M. x giganteus* and *A. donax* plants with fertilization rates ranging from 10 to 300 t ha^{-1} yr^{-1}, and found that biomass samples gave similar gases emissions in the combustion test.

Regardless of whether the nutrient demand of crops is provided by conventional fertilizer or waste residues, emission valorization in these studies is relatively scarce. This is the reason for conducting energy crop sewage sludge experiments that test plant growth along with ash constituent and incineration emission. The aims of this study are to 1) test the pollutant transfer from sewage sludge to energy crop *M. x giganteus* and *A. donax*, and 2) elaborate the ash constituents and exhaust gas emission characteristics of the feedstock materials. To reach these objectives, a pot experiment was carried out using two growth media; one based on soil with conventional fertilization and one directly on dewatered sewage sludge growth.

Materials and Methods

Experimental Details and Treatments

Dewatered sewage sludge was obtained from a municipal wastewater treatment plant located in Sakarya, Turkey. After an extended activated-sludge treatment process, thickened sludge was mechanically dewatered using a belt filter, generating a sludge cake of about 20% dry matter content in the treatment facility (Dede *et al*., 2015). Initial physicochemical characterization of the sewage sludge used in the experiments is shown in Table 1. A sandy clay loam soil was used in the study. The texture was sand (>0.2 mm) 25%, silt (0.002–0.2 mm) 40%, and clay (<0.002 mm) 35%. The detailed chemical properties of the experimental soil are given in the Table 1.

Plastic pots (each containing 10 kg of either soil or dewatered sludge) were used for the experiments. Identical-size (10 cm height) *M. x giganteus* and *A. donax* rhizomes were transplanted into pots (one in each) and the plants were allowed to grow for one growing season, from April to end of the October in 2016. The experiment was carried out under natural conditions with a mean daily temperature of 28°C and 13 h photoperiod. The planted pots were arranged in a completely randomized block design with four replications. Following four treatments, a total 16 plastic pots were installed.

Soil, Sludge and Plant Analysis

The soil and sludge samples were dried at 70°C until reaching a constant weight, and were ground and sieved with a 2 mm mesh size before being subjected to the physicochemical characterization analysis. pH and electrical conductivity (EC) were measured at the 1:5 (w/v) ratio of soil water suspension, using a pH and EC meter electrode (Ozdemir *et al*., 2017). Organic matter (OM) and organic carbon (TOC) were measured using the Walkley and Black method (Dede *et al*., 2015). Nitrogen (N) was determined by the Kjeldahl procedure (Dede *et al*., 2015). Phosphorus was measured by spectrophotometric methods (Pierzynski, 2000), and potassium was measured using ICP-OES (Spectro Arcos, Kleve, Germany) by employing the ammonium acetate procedure (Dede *et al*., 2015).

Dried samples (~250 mg) of soil and sewage sludge were digested in a microwave digestion system using 6 mL of HNO_3 (65%) and 1 mL of H_2O_2 (30%) acid mixture. Following the wet digestion, heavy metal contents and micronutrients were determined using an ICP-OES (Spectro Arcos, Kleve, Germany).

Aboveground plant samples of *M. x giganteus* and *A. donax* were oven dried at 78°C until reaching a constant weight. The moisture content was then determined gravimetrically using the oven drying method. Dry plant samples were ground into a fine powder, sieved through a 2 mm sieve and 250 mg subsamples were digested in a microwave digestion system (Sorisole-Bg Italy) using 6 mL of HNO_3 (65%) and 1 mL of H_2O_2 (30%) acid mixture (Dede *et al*., 2015). After wet digestion, elemental composition analysis was performed using an ICP-OES (Spectro Arcos, Kleve, Germany).

The contents of lignin, cellulose, and hemicellulose in feed-stocks were obtained following the method described in Van Soest *et al*. (1991) with a thermostable amylase pre-treatment using a Fibertec System (FOSS Tecator, Hillerød, Denmark). Following the neutral detergent fibre (NDF), acid detergent fiber (ADF), and acid detergent lignin (ADL) content determination, the cellulose and hemicellulose content was calculated as: Cellulose = ADF - ADL; Hemicellulose = NDF - ADF. Lignin was expressed as acid detergent lignin (ADL).

To evaluate energy yield, treatment samples were milled at 1 mm and oven dried at 60°C and then at 105°C for 24 h, respectively (Zema *et al*., 2012). Following the wet weight determination, the higher heating value (HHV) was measured directly on a 1 g pill in a bomb calorimeter (SDM, SN 3472) at the reference temperature (25°C). The lower heating value (LHV) was estimated using the HHV measurements and the moisture content of the feedstock samples as follows (Zema *et al*., 2012):

$$\mathrm{LHV} = \mathrm{HHV}\left(1 - \frac{MC}{1000}\right) - Ps\left(\frac{MC}{1000}\right)\Delta Hv$$

Where, *MC* represents moisture content at 60°C (g kg^{-1}), *Ps* is sample weight (1.00 ± 0.01 g) and ΔHv is the heat of water vaporization (2.54 MJ kg^{-1}).

Feedstock samples were pelleted before the combustion flue gas analysis. Pelleted samples were burned in a pilot scale fluidized bed combustion plant, which was designed for the combustion of biomass fuels. The flue gas composition was performed using a multi gas analyser (TESTO 350 M XL-454), continuously measuring CO_2, CO, SO_2, NO and NO_x. Ash collected from the combustion test was subjected to the ash constituent analysis as previously described for sewage sludge and plant samples.

Statistical Analysis

The plant growing experiments were performed in a completely randomized design with three replicates per treatment. The data collected for the soil, sludge and plant samples were statistically analyzed using one-way analysis of variance (ANOVA) to check for any difference between the means. Significant means were separated using an LSD test technique at a 5% probability level.

Results

Plants cultivated on sewage sludge produced significantly higher dry biomass compared with plants cultivated on natural soil with conventional fertilization (Table 2). *A. donax* plants were more responsive to sewage sludge than *M. x giganteus* plants. The dry biomass yield was slightly lower in the case of *M. x giganteus* cultivated on sewage sludge. The initial growth dynamics of the plants cultivated on natural soil was faster in comparison with those cultivated on sewage sludge. However, following the adaptation period, the plants cultivated on sludge showed apparent plant growth with respect to soil treatment. The maximum dry biomass yield was observed at 2.06 kg m^{-2} for plants cultivated on sludge treatments (Table 2). The mean values of the biomass obtained in both species were significantly lower in soil treatments, but was more apparent in *A. donax* plants.

Table 1: Initial physicochemical properties of the soil and sewage sludge

Parameter	Experimental soil	Dewatered sewage sludge
pH	7.2	6.7
EC (dS m^{-1})	0.45	1.97
Organic matter (%)	1.54	58
$CaCO_3$ (%)	11.32	-
Total N (%)	0.23	3.62
P (g kg^{-1})	18.02	31.50
K (g kg^{-1})	61.86	12.00
Ca (g kg^{-1})	6.22	7.20
Mg (g kg^{-1})	6.36	260
Fe (mg kg^{-1})	6.98	121
Mn (mg kg^{-1})	4.47	277
Cu (mg kg^{-1})	4.21	19
Zn (mg kg^{-1})	2.14	971
Ni (mg kg^{-1})	0.92	76
Cr (mg kg^{-1})	1.23	212
Cd (mg kg^{-1})	<0.05	3
Pb (mg kg^{-1})	<0.05	31

Table 2: Biomass yield, moisture content, ash content, heating values and element analysis of the energy crop species cultivated on natural soil and dewatered sewage sludge

Parameter	Plant species					
	M. x giganteus			*A. donax*		
	Soil	Sludge	Diff (%)	Soil	Sludge	Diff (%)
Dry biomass yield (kg m^{-2})	1.73	1.89	8.46	1.81	2.25	19.55
Moisture content (%)	11.62	11.43	1.63	12.83	11.74	8.49
Ash content (%)	7.96	7.47	6.15	3.76	3.23	14.10
Volatile matter (% at 900°C)	73.45	74.24	1.06	75.42	76.63	1.58
Fixed carbon (%)	18.59	18.31	1.50	19.82	19.15	3.38
Cellulose (%)	48.82	49.55	1.47	39.77	40.63	2.12
Hemicellulose (%)	27.15	26.76	1.44	23.54	24.29	3.09
Lignin (%)	10.94	10.63	2.83	16.48	16.82	2.02
LHV (MJ kg^{-1})	16.43	16.09	2.07	16.89	16.26	3.73
HHV (MJ kg^{-1})	17.95	17.62	1.84	17.31	18.05	4.10
C (%)	49.08	49.73	1.31	47.97	48.86	1.82
O (%)	36.59	37.67	2.87	40.53	39.28	3.08
H (%)	5.72	5.70	0.35	5.77	5.68	1.56
N (%)	0.49	0.53	9.30	0.99	1.02	2.94
S (%)	0.09	0.11	9.09	0.12	0.11	8.33

There were significant differences in the cell wall composition of *M. x giganteus* and *A. donax* plants, but the effect of sewage sludge was not significant for both crops (Table 2). Cellulose and hemicellulose contents dominated in *M. x giganteus* but lignin contents in *A. donax* plants were significantly higher than in *M. x giganteus*.

Energy content values of biomass feedstock harvested from the treatments are presented in Table 2. The dry basis energy values for *A. donax* were in the range of 17.31–18.05 MJ kg^{-1}, and were not significantly different from *M. x giganteus*, which were in the range of 17.55–17.62 MJ kg^{-1} for soil and sewage sludge treatments, respectively.

Sewage sludge, as a growing media, significantly affects both the content of macro nutrients and heavy metals in plant tissue compared to the natural soil (Table 3 and 4).

Table 3: Dry biomass macronutrients content (g kg^{-1}) of two energy crops cultivated on sewage sludge and natural soil

Treatment	K	P	Ca	Mg	Na	Cl
Sludge *Arundo*	2.92a	0.42a	1.71a	0.58a	0.06	0.13
Soil *Arundo*	1.94b	0.29b	0.89b	0.35b	0.05	0.11
Sludge *Miscanthus*	2.81a	0.48a	1.68a	0.55a	0.09	0.16
Soil *Miscanthus*	1.88b	0.34b	0.81b	0.26b	0.07	0.12
LSD 0.05	0.089	0.036	0.145	0.095	n.s.	n.s.

Mean values in same column that share the same letter are statistically similar based on LSD comparison test at a 95% confidence level, n.s. no significant effects

Table 4: Dry biomass heavy metals content (mg kg^{-1}) of two energy crops cultivated on sewage sludge and natural soil

Treatment	Cd	Cr	Cu	Ni	Pb	Zn
Sludge *Arundo*	1.12a	2.02a	6.21a	2.46b	2.53a	51.79a
Soil *Arundo*	0.38b	0.29b	1.27b	0.92c	0.19c	10.91c
Sludge *Miscanthus*	1.11a	1.71a	6.64a	3.05a	1.83b	43.65b
Soil *Miscanthus*	0.29b	0.38b	1.39b	0.81c	0.18c	11.22c
LSD(P<0.05)	0.330	0.811	0.975	1.241	0.872	4.860

Mean values in same column that share the same letter are statistically similar based on LSD comparison test at a 95% confidence level, n.s. no significant effects

Regarding the difference in nutrient content between sludge and soil treatments, the sludge caused a significant increase of macronutrient concentration in plant tissues in both plant species ($P<0.05$). The plant tissue concentrations of K, P, Ca and Mg in the *A. donax* and *M. x giganteus* were significantly higher in plants cultivated on sewage sludge than in the soil treatments. Conversely, the concentration of Na and Cl in plant tissue varied insignificantly in either plant species or growing media ($P>0.05$). Regardless of the sludge or natural soil, nitrogen, potassium and calcium concentrations in *A. donax* plants were significantly higher than in *M. x giganteus* plants, whilst *M. x giganteus*plants accumulated more phosphorus than *A. donax* plants.

The ash composition of *M. x giganteus* and *Arunda* biomass are presented in Table 5. It can be observed that Si is the dominant compound both in *M. x giganteus* and *A. donax* ashes however, the rate was 10% higher in *A. donax* plants regardless of growing media. K and Ca were secondary high concentration elements, giving similar results for both feedstock plants. The rest of the minerals in the ash samples were quite similar for all the plants and growing media. Similarly, sum of Na, Al, Fe, and Mn comprised less than 1% of the ash.

As seen from the result presented in Table 6, the sewage sludge treatment resulted in gradually increasing NO_x and SO_2 emissions for both plant species. Emissions of NO_x were positively correlated with feedstock nitrogen content; therefore, the rate was higher and significant for *A. donax* plants. On average, NO_x emissions for *M. x giganteus* and *A. donax* increased by 6 and 10% in the case of sludge treatment compared with those of natural soil, respectively.

Table 5: Ash compositions of the experimental species cultivated on natural soil and dewatered sewage sludge

Parameter	Plant species					
	M. x giganteus			*A. donax*		
	Soil	Sludge	Diff (%)	Soil	Sludge	Diff (%)
Si	24	23	4	33	33	0
K	7	7	0	8	9	1
Ca	9	9	0	8	7	1
Mg	3	0	0	2	2	0
P	3	4	0	1	1	0
Al	<1	<1	0	1	1	0
Na	<1	<1	0	<1	<1	0
Fe	<1	<1	0	<1	<1	0
Mn	<1	<1	0	<1	<1	0
S	<1	<1	0	<1	<1	0
O	46	47	2	47	46	2

Table 6: Gaseous emissions of the experimental species cultivated on natural soil and dewatered sewage sludge

Parameter	Plant species					
	M. x giganteus			*A. donax*		
	Soil	Sludge	Diff (%)	Soil	Sludge	Diff (%)
O_2 (%)	18.42	18.23	1.03	18.11	18.47	1.95
CO_2 (%)	2.45	2.37	3.26	2.51	2.43	3.18
CO (mg m^{-3})	1222	1189	2.70	1116	1172	3.12
NO (mg m^{-3})	148	152	2.63	157	166	5.42
NO_x (mg m^{-3})	158	169	6.51	160	177	9.60
SO_2 (mg m^{-3})	8	9	11.11	9	10	10.00

Discussion

To understand the beneficial and detrimental effects of sewage sludge to energy crops, *A. donax* and *M. x giganteus* plants were cultivated on sewage sludge and their dry biomass characteristics, ash and air emission had been analyzed comparing the plants cultivated on standard natural soil. The findings on dry biomass yield, plant composition and energy content indicated that nutrient rich sewage sludge could be effective in increasing combustible feedstock. The higher biomass yield observed for both crops cultivated on sewage sludge could be attributable to the nutrient-providing capacity of the sludge, which contains appreciably greater amounts of primary macro- and micro-nutrients (Table 1). These observations are in close agreement with those reported in other studies on energy crops amended by waste materials (Smith and Slater, 2010). Moreover, perennial energy crop biomass yields increase progressively until the full establishment of crops to optimize land cover by increasing plant density (Acaroglu and Aksoy, 2005). It is also expected that, the continuation of plant density and growth rate would be further increase the dry biomass yield in following season. Energy rich constituents cellulose, hemicelluloses and lignin contents are generally related to the plant species and nutrient source effects are limited in most of the cases determined for different energy crop species (Brosse *et al*., 2012). The biomass composition is consistent with that of the reported value that is 35–40% cellulose, 25–30% hemicellulose and

25–30% lignin (Vassilev *et al*., 2010). The difference in ranges may be due to the plant species, growing conditions, sampling time and analytical method.

Energy values of the feedstock are strongly influenced by carbon contents of biomass (Baxter *et al*., 2014). Considering the similar energy content of different species cultivated on either sewage sludge or natural soil seems more related to the similar carbon content of feedstock. Moreover, relatively small amounts of ash content determined in sludge grown plants could have also been responsible for insignificantly high carbon and energy content of those treatments.

The chemical properties of feedstock potentially influence the combustion of both air and gaseous emissions (Carroll *et al*., 2015). Feedstock K, Cl, N and S contents play an important role in affecting biomass combustion quality, particulate matter (PM) and gases emissions (Brosse *et al*., 2012). The N content of *A.donax* plants (1.01%) was significantly higher than that in *M. x giganteus* plants (0.51%). *Arundo* had significantly higher levels of K (0.24%), while its S (0.11%) and Cl content (0.01%) were not significantly different from the *M. x giganteus*. The concentrations of three PM-forming elements in *M. x giganteus* were 0.22% K, 0.10% S and 0.01% Cl. Irrespective of the plant species, significant concentrations of heavy metals were determined in both *A.donax* and *M. x giganteus* plant biomass cultivated on sewage sludge. In general, macronutrients and heavy metal concentrations were lower in soil-grown plants than in the sewage sludge treatments, but were within the reported range of this kind of experiment (Kolodziej *et al*., 2016; Ociepa-Kubicka *et al*., 2016).

Both concentration and composition of ash can affect combustion quality and especially the heating value of the biomass feedstock (Smith and Slater, 2010). Therefore, ash content and its mineral elements, such as P, K, S and Cl, are desired to be in lower concentrations for combustion efficiency and to prevent environmentally harmful emissions (Smith and Slater, 2010). The ash compositions of the crops are generally different due to species diversity, growing conditions, and growth amendments used. In the present study, the concentration of all studied elements in both crop species depended on the growing media. For instance, *M. x giganteus* and *A. donax* ash obtained from sewage sludge has a much higher Si, K and Ca content and lower Al and Fe contents than ashes obtained from plants cultivated on natural soil. It is also noticeable that, P content was quite high in the *M. x giganteus* ash compared with *A.donax*. The impact of waste materials on crop mineral content has been also reported for other biomass ash, such as barley straw and corn stalks (Kolodziej *et al*., 2016).

High heavy metal concentrations in the final solid emission ash are unwanted since they create disposal difficulties or constrain reuse for nutrients to the plants (Dede *et al*., 2015). As presented in Table 6, the heavy metal concentration in biomass ash increased with the use of sewage sludge as a growing media. The Pb, Ni and Cr values in ash were higher in the plants grown on sludge in comparison with natural soil. However, heavy metal concentrations in ash determined from the sludge treatments were in the range of reported values obtained by similar studies (Baxter *et al*., 2014).

The presence of N and S in biomass feedstock can result in emissions of NO_x and SO_2 (Brosse *et al*., 2012). There are studies reporting the correlations between biomass-bound N with NO_x emission (Carroll *et al*., 2015; Garcia-Maraver *et al*., 2015). In this regard, we for the first time explored that the nutrient transfer directly from nutrient rich sewage sludge to biomass feedstock and subsequent combustion emissions. There was no statistical evidence indicating the treatments were effected by air emissions. In this regard, measured O_2, CO_2 and CO emissions in the present study are in line with Garcia-Maraver *et al*. (2015), who attributed their observation to inherent oxygen content of biofuel and combustion temperatures that improved the oxidation of incomplete combustion and CO and CO_2 emissions. Despite the elevated tissue N in sewage sludge grown plants, NO_x emissions of both feedstocks were below the national and European standards of 300 mg Nm^{-3} (Villeneuve *et al*., 2012).

Conclusion

Sewage sludge stimulated plant growth and dry matter accumulation in both plant species, but the effect was greater for *A. donax*. Energy contents were not affected by the either plant species, nutrient source or the growing media. Macro-element and heavy metal content in plant dry matter increased with sludge treatment, although slight variations in element content was observed in the crop species. *A. donax* showed a tendency to accumulate both macro elements and heavy metals in dry feedstock. Si was the dominant compound in both species, but the rate was higher in *A. donax* plants regardless of growing media. Increasing N concentration in plant tissue provided by sewage sludge resulted in increasing NO_x in exhaust gas emissions. A maximum increase (~10%) in the NO_x emissions was with *A. donax* plants. Also, NO_x emissions with *M. x giganteus* were increased by sludge application, but the rate was slow. SO_2 emissions were consistent with plant species but emission was very small. Consequently, our result showed that sewage sludge could be effectively used as nutrient source for energy crops without deteriorating exhaust gas emissions.

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