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PHYSICOCHEMICAL AND BIOLOGICAL CHARACTERISATION OF THE SLUDGE FROM THE ROMANIAN MUNICIPAL WASTEWATER TREATMENT PLANTS FOR ASSESSMENT OF ITS CONFORMITY LEVEL WITH THE LEGAL NORMS FOR AGRICULTURAL USE

D. CIPRIAN*, C. COSMA, E. PENA-LEONTE, A. BALLO

National Research and Development Institute for Industrial Ecology – ECOIND, 90-92 Sos Panduri Street, Sector 5, Bucharest, Romania
E-mail: *ecoind@incdecoind*

Abstract. The primary and biological sludges from municipal wastewater treatment plants represent its main solid residue. Because of its high organic load and the content in nitrogen and phosphorus compounds, the sludge may be used in agriculture. In order to check the possibilities to use for application in agricultural lands, the anaerobic fermented sludge was characterised for the following physicochemical and bacteriological parameters: total N, P, heavy metals, fecal coliform. The analyses were conducted in compliance with the Romanian standard methods as follows: total N content varies between 36.7-44.6 g/kg d.s., from which NH_4^+–N represents ≤ 10%, and NO_3^-–N does not exceed 0.9%; P expressed as P_2O_5 is 1.2-2 g/kg d.s., and K evaluated as oxide (K_2O) presents values of 3-5 g/kg d.s; the analysed heavy metals have lower concentration values than the limits for a biological sludge used as a fertiliser in agriculture, in conformity with Directive 86/278 EEC; according to EPA, the municipal wastewater treatment plant residual sludge, treated by anaerobic fermentation, is included in B class sludge. Pathogens indicators analysed for this class of sludge are – the density of fecal coliform, indicator which can be analysed in the same manner as *Escherichia coli,* the most important pathogen agent from this category of microorganisms. According to the norms of EPA, a sludge which is going to be used as an agricultural fertiliser must contain a smaller number than 2 millions colonies formatting units (CFU) per gram of total solids in the moment of its use. The results were compared with the Norms stipulated by Decree 49/14 January/2004 for approval of the Technical norms concerning the protection of the environment and especially for the soil when the sludges are used in agriculture. The results show that the sludges from the Romanian Municipal WWTPs may be used as fertiliser for less sensitive culture like: textile plants and pasture field.

Keywords: WWTPs, sludge, agriculture.

AIMS AND BACKGROUND

The objective of this study is to determine the quality of two Romanian Municipal WWTP's waste sludge in accordance with the EU Sludge Directive 86/278/EEC (Ref. 1) and OMAPAM 49/2004 (Ref. 2) (Order for approval of the technical

* For correspondence.

norms regarding the protection of environment and especially of soil, when the sewage sludge are used in agriculture), in order to initialise the agricultural reuse of sewerage sludge.

The European Union has adopted various regulations in order to establish the recycle of organic waste as fertiliser, compost or soil amendment.

The first disposition in this topic is the Directive 86/278/EEC (Ref. 1) on the protection of the environment, when sewage sludge is used in agriculture in order to prevent harmful effects on plants, soils, animal and human beings as well as promoting sludge in a safe manner.

According to this Directive, sewage sludge must be treated before its agricultural use. One example of effective sludge treatment process is the anaerobic digestion that is applied in many municipal WWTPs.

At the moment, the European Commission is working on a new text for the Directive 86/278/EEC.

The main new areas of focus are (3rd draft, April 2000), as follows:

- some microbiological criteria for the control of hygienisation or stabilisation process;
- lower limit values for heavy metals and stricter limits in sewage sludge:

– in various heavy metals the maximum permitted amount is reduced by half,

– sewage sludge can not be used for soil at pH less than 5.0;

- limit values not only for heavy metals, but also for different groups of persistent organic compounds, such as PCBs, PAHs, dioxins, etc.;
- it requires different treatment for sludges depending on the types of land application to be done (as US EPA regulations stipulated).

EXPERIMENTAL

CHARACTERISATION OF THE FERMENTED CENTRIFUGED SLUDGE SAMPLES DROWN FROM THE WASTEWATER TREATMENT PLANTS (NUMBER OF HABITANTS BETWEEN 100 000-300 000)

Primary and secondary residual sludge, resulted from the municipal wastewaters treatment were pretreated as follows:

- centrifugal thickness;
- anaerobic fermentation;
- dewatering of the fermented sludge.

Fermented sludge ($\tau_{ferm.} \cong 21$ days, $t_{ferm.} = 30\text{-}35^{o}$) was dewatered through centrifugation in the presence of cationic polyelectrolyte. Fermented and dewatered sludge was carried out to an ecological waste dump, to be stored.

The utilisation of the centrifuged and fermented sludge as fertiliser in agriculture is limited by the toxic elements presence (especially heavy metals) that are regulated by the European and Romanian legislation (Directive 86/278 EEC;

Decree 49/2004 (Refs 1 and 2)). The level of pathogenic bacterial loading is limited also by the 3rd Draft/2002 of EU Sludge Directive 86/278/EEC (Ref. 1).

Analytical investigations aimed to evaluate the centrifuged-fermented sludge quality, produced by two wastewater treatment plants, and respectively the compliance with the requirements of European Directive 86/278 EEC.

To establish the characteristics of dewatered sludge composition from these two WWTPs, the following indicators classes were analysed:

- heavy metals: Cd, Cu, Ni, Pb, Zn, Hg, Cr and Mn;
- primary nutrients: P(P_2O_5), K(K_2O), N(N_t, N–NO_3^-, N–NH_4^+);
- secondary nutrients: Ca (CaO), Mg (MgO);
- bacterial loading: coliform bacteria, *Escherichia coli*;
- pH, humidity.

Characterisation data of the sludge samples are summarised in Tables 1 and 2.

Table 1. Characterisation of centrifuged sludge – NCC samples

Indicators	MU	Sludge samples		Max. permissible values		
		NCC1	NCC2	Directive 86/278/CE	3 Draft new directive	OMAPAM 49/2004
pH	pH unit	7.58	7.93	–	–	–
Humidity	%	70.3	74	–	–	–
N_{total}	mg/kg d.s.	40350	44600	–	–	–
NH^+_4–N	mg/kg d.s.	3400	3420	–	–	–
NO^-_3–N	mg/kg d.s.	370	240	–	–	–
P_2O_5	mg/kg d.s.	1400	1170	–	–	–
MgO	mg/kg d.s.	17900	8400	–	–	–
CaO	mg/kg d.s.	121800	54400	–	–	–
K_2O	mg/kg d.s.	2850	2200	–	–	–
Cd	mg/kg d.s.	< 0.5	< 0.5	20-40	10	10
Cu	mg/kg d.s.	95	12	1000-1750	1000	500
Ni	mg/kg d.s.	52	26	300-400	300	100
Pb	mg/kg d.s.	34	86	750-1200	750	300
Zn	mg/kg d.s.	1050	52	2500-4000	2500	2000
Hg	mg/kg d.s.	< 0.1	< 0.1	16-25	10	5
Cr	mg/kg d.s.	< 0.5	< 0.5	–	1000	500
Mn	mg/kg d.s.	300	130	–	–	–
Escherichia coli	CFU*/g	1329×10^3	1130×10^3	–	$< 5\times10^2$**	–
Coliform bacteria	No/g	1992×10^3	1830×10^3	–	–	–

*Colonies formatting units (max. for B class); ** imposed value, after sludge hygienisation.

Table 2. Characterisation of centrifuged sludge – NCM samples

Indicators	MU	Sludge samples		Max. permissible values		
		NCM1	NCM2	Directive 86/278/CE	3 Draft new directive	OMAPAM 49/2004
pH	unit pH	7.69	8.26	–	–	–
Humidity	%	69.25	72.77	–	–	–
N_{total}	mg/kg d.s.	36740	41860	–	–	–
NH_4^+–N	mg/kg d.s.	600	4530	–	–	–
NO_3^-–N	mg/kg d.s.	360	340	–	–	–
P_2O_5	mg/kg d.s.	2000	1400	–	–	–
MgO	mg/kg d.s.	17900	11800	–	–	–
CaO	mg/kg d.s.	69800	94300	–	–	–
K_2O	mg/kg d.s.	5000	3000	–	–	–
Cd	mg/kg d.s.	< 0.5	< 0.5	20-40	10	10
Cu	mg/kg d.s.	130	170	1000-1750	1000	500
Ni	mg/kg d.s.	52	33	300-400	300	100
Pb	mg/kg d.s.	450	123	750-1200	750	300
Zn	mg/kg d.s.	2130	350	2500-4000	2500	2.000
Hg	mg/kg d.s.	< 0.1	< 0.1	16-25	10	5
Cr	mg/kg d.s.	< 0.5	< 0.5	–	1000	500
Mn	mg/kg d.s.	0.024	0.017	–	–	–
Escherichia coli	CFU*/g	987×10^3	1192×10^3	–	$< 5\times10^2$**	–
Coliform bacteria	No/g	1860×10^3	1980×10^3	–	–	–

*Colonies formatting units (max. for B class); ** imposed value, after sludge hygienisation.

Analytical data are as follows:

• all the sludge samples have a slightly alkaline reaction, the pH varying in the range of 7.6-8.3;

• the content of dry substance (≤ 30%) is specific to the anaerobic fermented and dewatered sludge by centrifugation;

• the analysed elements, thought as primary and secondary nutrients, have different range values, which are accepted by literature:

– N_{total} content varies between 36.7-44.6 g/kg d.s., that is NH_4^+-N represents ≤ 10% from total N (NCC sludge: N–NH_4^+ = 7.7-8.4%; NCM sludge: N–NH_4^+ = 1.6-10%), and NO_3^-–N that does not exceed 0.9%;

– P expressed as P_2O_5 is 1.2-2 g/kg d.s., and K evaluated as oxide (K_2O) emphasises values of 2.2-2.8 g/kg d.s. for NCC sludge and respectively 3-5 g/kg d.s. for NCM sludge;

– secondary nutrients such as Ca(CaO) and Mg (MgO) take values between large limits:

– NCC sludge: CaO ≅ 54.4-121.8 g/kg d.s.; MgO ≅ 8.4-17.9 g/kg d.s.;
– NCM sludge: CaO ≅ 69.8-94.3 g/kg d.s.; MgO ≅ 11.8-17.9 g/kg d.s.;

• the analysed heavy metals have lower concentration values than the limits for a biological sludge used as a fertiliser in agriculture, in conformity with Directive 86/278 EEC and;

• Cd, Cr, Hg were not identified in the sludge samples, the concentration values being under the method limits;

• Cu and Ni concentrations are of tens mg/kg d.s. for both types of sludges;

• Pb concentrations varies from tens mg/kg d.s. (NCC samples) to hundreds mg/kg d.s. (NCM samples). According to the limits stipulated in the Romanian legislation, which are more strict, the exceeding of Pb value is possible to be registered in the case of NCM sludge;

• Zn concentrations varies in large limits for both types of sludge from tens mg/kg d.s/ to thousand mg/kg d.s.;

• Mn, a pollutant which is not regulated, was identified at concentration values of hundreds mg/kg d.s. only in the case of NCC samples.

The Romanian legislation does not point the limits of bacterial content for the waste sludge from wastewater treatment plants, which are going to be used as agricultural fertilisers. In consequence, the sludge bacterial content sampled from the wastewater treatment plants has been evaluated after 'EPA'S Part 503-Biosolids Rule' (Ref. 3).

According to EPA, the wastewater treatment plant residual sludge, treated by anaerobic fermentation, is in B class sludge, at which it is analysed as a pathogens indicator – the density of fecal coliform, an indicator which can be analysed in the same manner as *Escherichia coli*, the most important pathogen agent from this category of microorganisms. According to the norms of EPA, 'a sludge which is going to be used as an agricultural fertiliser must contain 7 samples as an average of a smaller number than 2 millions/g total solids or less than 2 millions colonies formatting units (CFU) per gram of total solids in the moment of its use' (Ref. 3).

CONCLUSIONS

The determined data give information concerning the composition and the quality of anaerobic fermented and dewatered sludge, resulted in the wastewater treatment plants, in order to assess its conformity level with the legal norms for agricultural use. The analysis of the main indicators, heavy metals and bacteriological load is compulsory:

• The concentration of heavy metals (Cd, Cu, Ni, Pb, Zn, Hg and Cr) were compared with the Norms stipulated by Decree 49/14 January/2004 for approval

of the technical norms concerning the protection of the environment and especially for the soil when the sludges are used in agriculture:

– the content of heavy metals (except Pb for one sample) is in accordance with the stipulated limits.

• Pathogens indicators analysed for this type of sludge is – the density of *Escherichia coli*, the most important pathogen agent from this category of microorganisms:

– according to the norms of EPA, a sludge which is going to be used as an agricultural fertiliser must contain a smaller number than 2 millions colonies formatting units (CFU) per gram of total solids in the moment of its use;

– in conformity with the results, the municipal wastewater treatment plant residual sludge, treated by anaerobic fermentation, is included in B class sludge according to the EPA norms;

– the analysed sludges from the Romanian Municipal WWTP's may be used as a fertiliser for less sensitive culture like: textile plants and pasture field.

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