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Modification of the Chemico-Physical Properties of Sewage Sludge towards its Application for Agricultural Purposes

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Strictly as per the compliance and regulations of:

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Rosik-Dulewska Cz [α], Nocoń K [σ] & Karwaczyńska U [ρ]

Abstract - Municipal sewage sludge belongs to organic waste rich in organic matter, nitrogen and phosphorus and at the same time poor in potassium content. From the environmental viewpoint, sewage sludge should be recovered as fertilizer i.e. utilised for land applications. However, in order to do so, the sludge cannot contain heavy metals in amounts above the permitted levels.The article presents the results of a research carried out to investigate the potential of utilizing sewage sludge of high fertilizing values and elevated zinc content for agricultural purposes by mixing it in appropriate ratio with fly ash from lignite and hard coal combustion and granulation. Data from tests on enriching the sludge-fly ash mixtures with potassium to improve its fertilizing properties are also presented. The research was carried out taking into account relevant regulations effective in the EU countries. The investigations proved that sewage sludge can be successfully mixed with fly ash in weight proportion 1:1 and granulated while maintaining the minimum content of organic matter, nitrogen and phosphorus required for organic-mineral fertilizers and meeting the level of heavy metals permissible for sludge used for agricultural purposes. However, due to low potassium content in sludge-fly ash mixtures, an addition of potassium salt is proposed at maintaining simultaneously the proportion of sludge to ash as 7:3. The procedure for utilizing sewage sludge, a problem-causing organic waste, described in the paper shows that it is possible to produce an organic-mineral product of desired chemical properties for soil conditioning which through the granulation process acquires also a physical form and mechanical properties making it easy to transport and apply.

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I. Introduction

Management of the sewage sludge produced in increasing volumes from year to year due to sanitation of urban and rural settlements poses a serious challenge. The problem is not only the volume of the sludge itself but its quality as well. Due to the soil-forming and fertilizing values of sewage sludge resulting from high content of nitrogen, phosphorus, magnesium, calcium and microelements, its land application seems one of the most efficient solutions [13]. At the same time however, an increasing awareness of risks posed by introducing sewage sludge into the soil environment evokes serious concerns among scientists and farmers. For several years there has been a tendency in Europe to strengthen the regulations allowing for sewage sludge application to the soil environment to better protect it. The EU proposed an amendment of the Directive 86/278/EEC of 12 June 1986 on the protection of the environment and the soils when using sewage sludge (biosolids) as a fertilizer [2]. In 2000 a third draft of the working document concerning this amendment was issued (ENV.E.3/LM) [15]. The changes proposed in the sludge directive refer mainly to more stringent requirements concerning heavy metals content (Table 1) and introduction of a monitoring of organic pollutants. The document indicates the need to extend the scope of the effective legal regulations and performance of sewage sludge management in such areas as forests, green areas and areas for reclamation. It states that sewage sludge should find application whenever an increase of yields or soil fertilization are needed. When applying sludge, other legal acts should be also taken account of, especially Directive 91/676/EEC concerning water protection against pollution caused by nitrates originating from agricultural sources. Manner in which the sewage sludge is utilized should ensure a minimal negative effect on human and animal health, plants, quality of ground and surface water; soil quality in a long-time perspective and biodiversity [1]. Due to planned restrictions concerning permissible heavy metals content in sewage sludge introduced to the soil, more restrictions should be expected in the stabilized sludge application for agricultural purposes, especially in the case of sludge originating from municipal and industrial wastewater treatment plants that contains high amounts of heavy metals which are not categorised as plant nutrients (mercury, lead, cadmium). Sewage sludge containing excessive amounts of these heavy metals which are indispensable for plants can be used for production of microfertilising preparations and enrichment of basic fertilisers in microelements (e.g. zinc, copper, manganese, iron). Reduction of heavy metals concentration can be achieved by adding a foreign matter to the sludge e.g. an addition of plant mass

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followed by composting of both components. Also liming is an example of heavy metals concentration reduction in sludge [14]. Another practice consists in mixing sewage sludge with fly ash, especially from lignite, which is rich in calcium compounds. However, its should be underlined that mixing sewage sludge with lime or fly ash reduces the content of organic matter and the fertilising components in a dry unit of the product. Nevertheless, sludge mixing with fly ash has a number of benefits. The binding properties of ash allow for a good solidification with sludge. The alcalescent properties of ash and its fine-grain form enable eliminating the risks posed by pathogens contained in sludge and reduce odour emission. Also a sanitation effect of the fly ash admixture to the sludge is known which reduces the washout of chemical contaminants while improving the physical and mechanical properties of the sludge [3,4,10,11,12]. The use of a special process line with drum, plate or matrix granulator allows achieving an appropriate form, size and mechanical stability of the sludge-ash mixtures which make their transport and application easier [3,8]. The goal of the research presented in this paper was to modify the physico-chemical properties of a stabilised sewage sludge with zinc content exceeding the permissible level for agricultural sludge application in such a way as to achieve a product which could be used in agriculture.

II. Materials and Methods

The following materials and components were used in the tests: (i) stabilised sewage sludge originating from the Zabrze-Śródmieście wastewater treatment plant (ii) fly ash from lignite combustion originating from the Bełchatów power plant and fly ash from hard coal combustion originating from the Ł aziska power plant (iii) hydrated lime (iiii) potassium chloride of potassium content of 60% recalculated to k_2o. the modification of the physico-chemical properties of the sewage sludge was carried out in the following way:

1. Fly ash was added to a pre-dried sewage sludge of a 50% dry matter content. Next the mixture was homogenised and granulated in a plate granulator with a regulated rotation speed and plate angle of 1m. The produced granulate was dried to the dry mater content level of ca 98%. Three granulates were prepared: N9 with lignite fly ash and sewage sludge content in weight proportion as 7:3, N10 with lignite fly ash and sewage sludge content in weight proportion as 1:1, and N14 with hard coal fly ash and sewage sludge content in weight proportion as 3:7.

2. Sewage sludge pre-dried to dry matter content of ca 50% was mixed with fly ash and hydrated lime, then granulated in a granulator with ring matrix of 8mm aperture diameter. The obtained granulate was dried to the dry matter content of ca. 95%. Two granulates were prepared: N41 with lignite fly ash and N42 with hard coal fly ash. The weight ratio of ash to sludge in both granulates was as 7:3, while the amount of the added hydrated lime was 3,3% by weight of the ash dry mass.

3. Sewage sludge pre-dried to the dry mass content of ca. 50% was mixed with potassium chloride, then with fly ash and hydrated lime. Next, the procedure was similar as in point 2. Two granulates were prepared: N41/m with lignite ash content and N42/m with hard coal ash content. The weight ratio of sludge to ash in both granulates was as 7:3, while the amount of hydrated lime was 3,3% by weight of the ash dry mass and the potassium chloride was 300 g/km of dry mass of the other components.

The contents of organic matter, nitrogen, phosphorus, potassium and heavy metals were determined in the investigated sewage sludge, fly ash and in the granulates. The chemical composition of the fly ash was determined applying the procedure of an accredited Laboratory of Carbochemistry IChPW Q/ZK/P/15/01/B „Determination of the chemical composition of ash by roentgen fluorescence" using Philips energy dispersive MiniPal spectrometer and the norm PN-G-04528-10:1998 "Solid fuels. Determination of the chemical composition of ash. Determination of disodium oxide (Na_2O) and dipotassium oxide (K_2O)" using flame photometry on BIOTECH Management AFP spectrometer. The content of organic matter in sludge and granulates was determined according to the norm PN-75/C-04616.01 "Water and wastewater. Special sludge tests. Determination of water, dry mass, organic substance and mineral substance in sewage sludge".

The content of Kjeldahl total nitrogen in the sludge and granulates was determined according to the norm PN-75/C-04576.17 "Water and wastewater. Investigation of nitrogen compounds content. Determination of Kjeldahl total nitrogen in sewage sludge". The content of phosphates in the sludge and granulates was determined according to the norm PN-75/C-04537/14 "Water and wastewater. Investigation of phosphorus compounds content. Determination of total phosphates in sewage sludge by titration". Potassium content in the granulates modified with potassium salt was determined according to the norm PN-75/C-04591.05 "Water and wastewater. Investigation of potassium ion content. Determination of potassium ion content in sewage sludge by weight method". To determine the total content of metals: copper, zinc, nickel, lead, cadmium, chromium and potassium in the sludge and granulates, atomic absorption spectroscopy (AAS) was applied using UNICAM –PHILIPS PU9100 X spectrometer. Following the reference method as specified in the Regulation on municipal sewage sludge, prior to the determination the samples were extracted in aqua regia. [6]. Mercury content was determined in air dry samples according to the methodology recommended by the Polish Geological Institute using AMA 254 mercury analyser.

III. Research, Results and Discussion

The sludge used for the tests originated from a mechanical-biological wastewater treatment plant located in an industrial part of an urban agglomeration. Considering its land application, in particular for agricultural purposes, some limitations may appear, mainly due to a significant content of one or more heavy metals in the sludge mass. Typically however, the composition of sewage sludge undergoes seasonal changes resulting from the quality of the influent. The properties of the sewage sludge applied in the described research are presented in Table 2 and 3. The sewage sludge was rich in organic matter, its content amounted to 58% d.m. The content of nitrogen and phosphorus in the sludge was on the medium level of these biogenes content determined in sludge from municipal wastewater treatment plants in Poland [5], and amounted 3,6% d.m and 2,9 % d.m., respectively. Typically for municipal sewage sludge, the content of potassium was low i.e. 0,21 % d.m. Among metals contained in the sludge zinc was dominant. Following the Regulation [6], its content on the level of 3567 mg/kg d.m. allows for sludge application for specific needs resulting from the waste management plans, spatial development plans or decisions concerning building conditions and land management, for plants cultivation for composting purposes as well as purposes other than consumption or fodder production. At the same time the content of other heavy metals in the sludge was low, which would enable its agricultural application. To extend the scope of its potential land applications, the sludge was mixed with fly ash from coal combustion. Fly ash admixture to the sludge enables reduction of heavy metals concentration through the so called heavy metals dilution process, so that they are no longer a barrier in sludge application for agricultural purposes. Two types of fly ash were used in the investigations, their selected properties are presented in Table 2 and 3. Coal combustion processes taking place in the furnace cause that trace amounts of organic matter and nitrogen occur in fly ash. The content of phosphorus in fly ash from hard coal was higher compared to its content in the fly ash from lignite, whereas potassium content in the fly ash from lignite was comparable to its content in the sludge and nearly ten times higher in the fly ash from hard coal. Except for copper, the content of the determined metals was higher in the fly ash from hard coal combustion compared to the corresponding values in the fly ash from lignite. Comparing the content of heavy metals in the fly ash and in the sludge, higher values for nickel and chromium were determined in the sludge, however metals from the fly ash were extracted with a stronger extractant (see the section on methodology). It should be thus expected, that the tested mixtures of sewage sludge and fly ash will be characterised by lower fertilising values and higher nickel and chromium content than the sludge alone. These differences will increase with a raising content of fly ash in the mixtures. Simultaneously however, higher amounts of fly ash added to the sludge allow achieving a beneficial effect of the reduced zinc content i.e. the metal dominant in the sludge. However, due to the adopted assumption that the final product (sludge-ash granulate) should meet the effective legal regulations for land application, the content of the fertilising components should be in line with the requirements specified by the Regulation of the Ministry and Rural Development on the execution of selected provisions of the Act on Fertilising and Fertilisers [7] as well as the requirements of the Regulation of the Minister of Environment on the municipal sewage sludge [6]. An additional issue discussed by the authors in another publication [9] was to obtain a product of appropriate physical qualities (granulation and mechanical durability) to enable its storage, transport and application. Tables 4 and 5 present the data from the tests as described in the section „Materials and methods" on the fertilising qualities and standardised heavy metals content of the granulated sludge-ash mixes. Sludge is the component that impacts the fertilising quality of the granulate. Therefore these mixtures that contained the highest amount of sludge i.e. N14, N41 and A 42 (sludge to ash weight ratio as 7:3) showed the highest contribution of the organic matter (41,2-43,5%), total nitrogen (2,32-2,41%) and phosphorus in oxide form (5,18-6,09%). The fertilising qualities of the other granulates were lower, nevertheless still on the level satisfying the requirements for organic-mineral fertilizers set up in the Regulation on executing selected provisions of the Act on Fertilising and Fertilisers (Table 3) [7]. The exception was the content of organic matter in granulate N9 (sludge to ash weight ratio as 3:7) which was 20,3%. Granulates composed only of sludge and fly ash (N9, N10, N14, N41 and N42) contained low amounts of potassium i.e. from 0,18 to 0,42 % K_2O, while the required minimum content for organic-mineral fertilizers is 1%. Therefore a decision was made to enrich the mixtures with potassium in a form easily available for plants. After KCl addition to the generic mixtures, the achieved potassium content was on the level of 8,43% for N41/m granulate and 8,83 % for N42/m granulate and was significantly higher than the required 1%. Moreover, the addition of potassium resulted in an increase of the percentage ratio of potassium to phosphorus (oxide forms) to the value of 2,4. A high percentage ratio of these elements should ensure their successive availability to plants; potassium being released faster due to its better solubility in water solution. An analysis of the data from heavy metals content determination in granulates with no potassium salt addition showed that along with an increasing contribution of sewage sludge in the dry mass of the granulate the zinc and lead content raised while the nickel content dropped. Comparing the granulates with similar share of sludge and ash but where ash was of different origin i.e. granulates N41 with N42 and N41/m with N42/m, it can be stated, that the granulates with hard coal ash addition contained more zinc, nickel and chromium and less copper than the granulates with lignite ash addition. Copper content in granulates without potassium salt addition was in the range of 116 mg/kg d.m. (N42) to 146,37 mg/kg d.m. (N14) while in granulates enriched with potassium it was lower, amounting to the values of 82 mg/kg d.m (N42/m) and 92 mg/kg d.m (N41/m). These values were significantly lower than the permissible copper content in sewage sludge for land applications and in organic-mineral fertilisers. The content of zinc in the granulates was in the range of 1340,3 mg/ kg d.m. (N 9) - 2752 mg/kg d.m. (N14). The permissible content of 2500 mg Zn/kg d.m. for sewage sludge for agricultural applications was exceeded only in the case of granulate N14. Cadmium content in the granulates was in the range of 2,20 mg/kg d.m. - 3 mg/kg d.m. and did not exceed the permissible level of 10 mg/kg d.m specified in Regulation [6]. Granulates without potassium salt addition contained nickel in the amounts ranging from 32,2 mg/kg d.m. (N41) to 46,17 mg/kg d.m. (N9) while in granulates with potassium salt addition, nickel content was lower, on the level of 23,1 mg/kg d.m (N42/m) and 20,2 mg/kg d.m (N41/m). These values were much below the permissible content values defined for sewage sludge for land applications. Lead content in the granulates was in the range of 126,3 mg/ kg d.m. (N 9) to 207,5 mg/kg d.m. (N14). The permissible lead content of 500 mg/kg d.m. for sewage sludge used for agricultural purposes was not exceeded. The content of chromium determined in the granulates ranged from 42,4 mg/ kg d.m. (N 41/m) to 70,37 mg/kg d.m. (N10) and was below the permissible chromium content in sludge used for land applications and in organic-mineral fertilizers. Mercury in the granulates occurred in the amounts ranging from 0,95 mg/kg d.m. (N 9) to 1,71 mg/kg d.m (N 14) and did not exceed the permissible values. An overall analysis of the heavy metals content data obtained for the granulates against the permissible content values specified for sludge for different land applications in Regulation [6], shows that the granulated sludge-ash mixtures N9, N10, N41 and N42 and the two with potassium salt addition i.e. N41/m and N42/m, meet the requirements for sludge to be used for different land applications. In the case of the N14 granulate, an exceeded value of zinc content was observed for sludge used for agricultural purposes which means that it can find application for reclamation purposes of land other than agricultural i.e. for adopting the land for specific needs resulting from the waste management plans, spatial development plans or decisions on the building conditions and land management as well as cultivation of plants for composting purposes, purposes other than consumption and fodder production. A comparison of data on heavy metals content in the investigated granulates with the values specified in the Regulation on the execution of selected provisions of the Act on Fertilisers and Fertilising [7] shows that the permissible values for zinc and lead content were exceeded in all granulates except for N9 which contained the lowest amount of sewage sludge. Thus, composing sludge-ash mixtures means compromising between a product of high fertilising qualities (i.e. maximising the sewage sludge content in the mixture) that at the same time has to meet the requirements of heavy metals content specified by effective legal regulations. However, in the investigated granulates, an improvement of the fertilizing qualities occurred with an increase of the sewage sludge content in the dry mass of the mixture which in consequence caused also elevated zinc and lead amounts i.e. metals dominating in the sludge. The tests of the granulated mixtures of a selected sewage sludge and fly ash from hard coal and lignite prepared in three different proportions of these components (3:7, 1:1, 7:3), showed that the following granulates met the targeted effect of the research i.e. obtaining a product of required fertilizing qualities and heavy metals content below the permissible level for sewage sludge applications for agricultural purposes: N10 (sludge to ash ratio 1:1), N41 and N42 (sludge to ash ratio 7:3). These granulates can be considered substitutes for two-compound NP organic-mineral fertilizers. The equivalents of granulates N41 and N42 modified with potassium salt (N41/m and N42/m) contained by 30% less organic matter, nitrogen and phosphorus but higher amount of potassium (over 8% of K_2O). These granulates can be considered substitutes for three-compound NPK organic-mineral fertilizers.

IV. Summary

There are many situations in the practice of managing sewage sludge from industrial agglomerations when the heavy metals content exceeding the permissible standards becomes the main barrier in applying the stabilised sludge for agricultural purposes. One of the known and worth considering solutions to overcome this barrier is sludge mixing with fly ash – a byproduct which also finds application in agriculture e.g. as fertilisers components or for land reclamation. An additional advantage of the procedure is the opportunity to modify the chemical properties (including fertilising qualities) by selecting an appropriate proportion of the components as well as the physical and mechanical parameters by granulating the sludge-ash mixtures. In each case however, a compromise should be reached between the fertilising quality of the products and the heavy metals content so as to meet the legal requirements set up for waste used for agricultural purposes as well as to achieve a positive environmental effect. Using as an example a selected sewage sludge (with zinc content above the permissible level for sludge to be used in agriculture) and fly ash from lignite and hard coal, the authors of the presented research proved that these types of waste can be successfully mixed in proportions 1:1 – 7:3 and granulated while meeting the permissible heavy metals levels for sludge used for agricultural purposes together with the minimum content of organic matter, nitrogen and phosphorus required for the organic-mineral fertilizers. Nevertheless, neither sewage sludge nor granulates made exclusively of sludge and ash can be considered a sufficient source of potassium for plants. The investigations showed that sludge-ash mixtures can become a complete product for soil conditioning only after their enrichment with potassium fertilizer (KCl), a source of potassium easily available for plants. According to the authors it is beneficial to apply the weight proportion of the sludge-ash mixture components as 7:3 with its simultaneous modification with potassium salt. Since the sewage sludge is a dominant component of the granulate (70% by weight), the quality of the granulates from the viewpoint of heavy metals content will depend primarily on the sludge quality. The composition of sludge from a given wastewater treatment plant, including the content of heavy metals, is subject to seasonal changes due to the volume of the influent. In order to ensure the heavy metals content on the level permitted by law, the verification should be carried out already at the stage of sewage sludge selection. Assuming that the elemental composition of the fly ash does not undergo significant changes and the ratio of sludge to ash is as 7:3, the metal content in the sludge should not be higher than (MeP-0,3MeA)/0,7 , where MeP is the permissible metal content in the dry mass of sludge and MeA is the metal content in fly ash. The procedure for handling sewage sludge, a type of organic waste which poses challenges in its management, proves that it can be successfully utilised for production of an organic-mineral means for soil conditioning not only of desired chemical properties but of appropriate physical and mechanical qualities enabling its easy transport and application.

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Table 1 : Border values of heavy metals concentrations in sewage sludge to be used in soils: effective, proposed and prospective [mg/kg d.m.]

No	Metal	Directive 86/278/EEC	Proposed	Medium-term (ca.2015)	Long-term (ca. 2025)
1	Cu	1000-1750	1000	800	600
2	Zn	2500-4000	2500	2000	1500
3	Cd	20-40	10	5	2
4	Ni	300-400	300	200	100
5	Pb	750-1200	750	500	200
6	Cr	1000-1750	1000	800	600
7	Hg	16-25	10	5	2

Table 2 : The fertilizing qualities of swage sludge and fly ash

Contents [% dry mass]	Sludge	Fly ash	
		From lignite	From hard coal
Organic substance	58,13	Not determined	Not determined
Total nitrogen	3,60	None	None
Phosphorus	2,90	0,26	0,67
Potassium	0,21	0,21	3,06

Table 3 : Heavy metals content in sludge and fly ash [mg/kg d.m.]

No.	Metal	Sludge	Fly ash from hard coal	Fly ash from lignite
1	Cu	155,7	78	96
2	Zn	3569,3	208	125
3	Cd	3,1	<2	<2
4	Ni	20,7	107	48
5	Pb	187,1	151	113
6	Cr	36,9	163	126
7	Hg	1,99	Not determined	Not determined

Table 4 : The content of organic matter and biogenes in the granulates

Granulate	Type of fly ash	Weigh ratio of d.m. sludge:ash	Organic matter	K_2O	N_{total}	P_2O_5	K_2O / P_2O_5
			%	%	%	%	-
N 9	L	3:7	20,30	0,18	1,12	2,50	0,072
N 10	L	1:1	30,67	0,22	1,30	3,05	0,072
N 14	HC	7:3	41,20	0,41	2,41	5,18	0,079
N 41	L	7:3	43,56	0,40	2,38	5,89	0,068
N 42	HC	7:3	41,83	0,42	2,32	6,09	0,069
N 41/m	L	7:3	35,73	8,43	1,70	3,62	2,33
N 42/m	HC	7:3	36,36	8,83	1,67	3,76	2,44
organic-mineral fertilisers according to Regulation [7]			at least 30	at least 1	at least 1	at least 0,5	-

L- fly ash from lignite
HC _ fly ash from hard coal

Table 5 : Total heavy metal content [mg/kg d.m.],according to the method with aqua regia, in sludge-ash granulates vs. the permissible content values set up in the Regulation on municipal sewage sludge [6] and Regulation on the execution of selected provisions of the Act on Fertilising and Fertilisers [7]

	Metal [mg/kg d.m]						
	Cu	Zn	Cd	Ni	Pb	Cr	Hg
N 9	151	1340	3,0	46,1	126	63,5	0,95
N 10	117	2044	3,0	40,2	162	70,3	1,19
N 14	146	2752	3,0	39,2	207	65,5	1,71
N 41	128	2300	2,8	32,2	180	53,2	n.d
N 42	116	2370	2,9	38,0	184	61,8	n.d
N 41/m	92	1740	2,2	20,2	139	42,4	1,23
N 42/m	82	1748	2,2	23,1	128	57,2	1,23
Permissible content according to [6]	800	2500	10	100	500	500	5
Permissible content according to [7]	400	1500	3	30	100	100	2

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