

# The use of industrial waste and municipal sludge for the production of fertilisation pellets

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## Introduction

High prices for mineral fertilisers prompt the search for cheap, environmentally safe sources of organic matter and nutrients for plants that would produce economically sound effects.

It has been pointed out that certain municipal and industrial waste may be used for fertilisation. Studies have been undertaken on direct introduction of such waste into soils and processing thereof into soil conditioners and organic and mineral fertilisers that would comply with the norms set forth in the Regulation of the Minister of Agriculture and Rural Development (Journal of Laws of 2008 No. 119, item 765).

Studies carried out thus far on the potential use of municipal and certain industrial sewage sludges indicate that those sludges may be used for soil conditioning with organic matter and nutrients for plants. Municipal sewage sludges may contain excessive quantities of heavy metals, *Salmonella* bacteria, as well as intestinal parasites and their ova. Therefore, municipal sewage sludge should undergo hygienisation and stabilisation. Among numerous methods of stabilisation and hygienisation of sewage sludges, composting is used most frequently. A number of experiments have shown the positive impact of unprocessed and processed municipal sewage sludges on the increase of plant yield, improvement of quality indicators thereof, as well as improvement of soil fertility (Baran 2004 & 2005 [1, 2], Baran & Turski 2005 [3], Bernacka & Pawłowska 1994 [4], Czekala 2000 & 2004 [5,6], Drab et al. 2004 [7], Gambuś 1999 [9], Jędrzak 1998 [10], Kalembasa et al. 1999 [12], Kalembasa 2003 [13], Karoń & Pietr 2006 [14], Krzywy & Wołoszyk 1996 [15], Krzywy et al. 1998, 2000, 2002 [16, 17, 19], Krzywy & Jakubowski 2000 [18], Łabętowicz 2011 [21], Oleszkiewicz 1998 [22], Rosik-Dulewska 2008 [25], Siuta 2000 [28], Siuta & Wasiak 2001 [29], Urbaniak & Morzycka 1996 [30], Wołoszyk et al. 2000 [31]). The components for producing mixtures and then fertilisation pellets were chosen according to their chemical characteristics (content of nutrients for plants and certain heavy metals), microbiological characteristics (presence of *Salmonella* bacteria and ova of intestinal parasites), and the capacity to reduce the volume of waste in the environment and produce economically sound effects.

During production processes in ZCh POLICE S.A. the industrial sewage sludge is created. The analysis of chemical composition of the industrial sewage sludge has indicated that its reaction is neutral and that it contains organic matter and nutrients for plants (N, P, K, Ca, Mg and S) (Krzywy et al. 2012 [20]).

During the production of titanium dioxide (titanium white), iron (II) sulphate (VI) 7-hydrate is formed. Studies on potential use of iron (II) sulphate (VI) 7-hydrate as fertiliser have shown that it has positive impact on yield volume and improves yield quality. This waste contains sulphur and iron, thus increasing the volume of those elements in soils and plants (Frankenstein 2001 [8], Kacalski et al. 1998 [11], Krzywy et al. 1998 [16], Krzywy & Jakubowski 2001 [18], Pasikowski 2000 [23], Rozmarynowska 2009 [24], Wołoszyk et al. 2000 [31]).

The studies were aimed at creating four organic and mineral fertilisation pellet types and evaluating them in terms of fertilisation value.

## Scope and methods of study

The mixtures and subsequent fertilisation pellets were created using: municipal sewage sludge from wastewater treatment plant in Stargard Szczeciński, industrial sewage sludge from plant wastewater treatment facility in ZCh POLICE S.A., iron (II) sulphate (VI) 7-hydrate (a by-product from ZCh POLICE S.A.), mixture of sawdust from deciduous and coniferous trees (in 1:1 ratio), and urea. The components for production of mixtures and subsequent fertilisation pellets undergone chemical analysis (Tab. 1). The obtained results of microbiological tests of the municipal sewage sludge from the wastewater treatment plant in Stargard Szczeciński and the industrial sewage sludge from the wastewater treatment facility in ZCh POLICE S.A. indicated that the sludges did not contain *Salmonella* bacteria or ova of intestine parasites *Ascaris sp.*, *Trichuris sp.*, and *Toxocara sp.*

On the basis of the obtained test results, the material composition of individual fertiliser mixtures was developed (Tab. 2). In the production of fertiliser mixtures, the municipal and industrial sewage sludge and the mixture of deciduous and coniferous sawdust were applied in natural form. Iron (II) sulphate (VI) 7-hydrate and urea were used in the form of saturated aqueous solution by dissolving those substances at 18°C. The produced fertiliser mixtures were placed in plastic containers and stored for 48 h at 15°C. Next, the fertiliser mixtures were dried at 90°C, until agglomerates were formed. Agglomerates were minced into dust. Sodium lignosulphonate was then added in the amount of 2% of the weight of the mixtures. Fertiliser mixtures were sprinkled with water and granulated. The method was applied to verify the capability of forming pellets. The obtained fertilisation pellets were chemically analysed in terms of their suitability as fertilisers.

Chemical analysis of components and fertilisation pellets were conducted with the following methods: The pH H<sub>2</sub>O value acc. to PN-ISO 10390; dry matter acc. to PN-75C-04616/01; total nitrogen using Kjeldahl distillation method after mineralisation in concentrated sulphuric (VI) acid acc. to PN-ISO 11261; total phosphorus acc. to PN-98/C-04534-14; total potassium, magnesium and calcium using flame spectrophotometry acc. to PN-ISO 9964 1 & 2 on Perkin Elmer AAS300 spectrophotometer; total sulphur using nephelometric method; organic carbon using Lichterfelde method in Altena modification; total cadmium, copper, nickel, lead and zinc acc. to PN-ISO 02/8288.

The chemical analysis of iron (II) sulphur (VI) 7-hydrate was performed in the laboratory of ZCh POLICE S.A. in accordance with applicable Polish Norms. The statistical analysis of results related to fertilisation pellets was performed with ANOVA for single factor for Split-block design. Confidence half-intervals were calculated for  $p=0.05$ , using Tuckey's test.

## Results

Table 1 lists the chemical properties of waste used to produce the organic and mineral mixtures. Analysis of data in Table 1 indicates that:

- the highest pH H<sub>2</sub>O value was exhibited by industrial sewage sludge (8.80), followed by municipal sewage sludge (7.30). The lowest pH H<sub>2</sub>O value was exhibited by iron (II) sulphate (VI) 7-hydrate

- the highest dry matter content was found in sawdust (79.6%), followed by industrial sewage sludge (28.2%) and municipal sewage sludge (21.8%)
- sawdust and municipal sewage sludge contained considerably higher quantity of organic carbon (445 and 415 g·kg<sup>-1</sup> of dry matter), as compared to industrial sewage sludge (16.2 g·kg<sup>-1</sup> of dry matter)
- the highest level of macroelements (excluding sulphur) and certain microelements, including heavy metals, was found in municipal sewage sludge; Sawdust contained markedly less nutrients for plants and heavy metals; mercury was found in individual components in trace amounts
- the highest sulphur levels were found in iron (II) sulphate (VI) 7-hydrate (127 g·kg<sup>-1</sup> of dry matter).

Heavy metal levels in municipal sewage sludge did not exceed the threshold levels set forth in the Regulation of the Minister of Environment (Journal of Laws of 2010 No. 137, item 924), permitting the use of the sludge in agriculture and land recultivation. Heavy metal levels in other components used in the production of organic and mineral mixtures (excluding zinc levels in industrial sewage sludge) was lower than in municipal sewage sludge. Those data were taken into consideration in the development of material composition of fertiliser mixtures. The proportions of individual components were selected so that the chemical compositions of the obtained pellets would comply with the norms applicable to organic and mineral fertilisers.

**Table 1**
**Chemical properties of components used in production of organic and mineral mixtures**

Assay or quantification type	Component			
	Municipal sewage sludge	Industrial sewage sludge	Sawdust	Iron (II) sulphate (VI) 7-hydrate
pHH <sub>2</sub> O at 18°C	7.3	8.8	n/a	3.0
Dry matter, %	21.8	28.2	79.6	81.0
Total levels, g·kg <sup>-1</sup> of dry matter				
Organic C	415	16.20	445	none
Total nitrogen (N)	42.70	1.20	2.85	none
Phosphorus (P)	21.50	6.62	0.52	none
Potassium (K)	4.80	1.10	0.35	none
Calcium (Ca)	23.00	15.60	0.85	0.02
Magnesium (Mg)	2.80	1.69	0.95	0.29
Sulphur (S)	6.10	1.80	0.16	127
General levels, mg·kg <sup>-1</sup> of dry matter				
Cadmium (Cd)	3.25	0.87	0.17	0.28
Chromium (Cr)	57.00	8.75	1.77	none
Copper (Cu)	127	21.0	4.22	0.42
Nickel (Ni)	25.20	1.52	0.90	0.06
Lead (Pb)	62.50	50.4	2.25	1.98
Zinc (Zn)	1125	115	40.20	4.33
Mercury (Hg)	trace	trace	trace	trace

Abbreviations: n/a – not analysed

The obtained results conform to the studies by Baran & Turski (1999) [3], Bernacka & Pawłowska (1994) [4], Czekala (2000 & 2004) [5, 6], Drab et al. (2004) [7], Gambuś (1999) [9], Kacalski et al. (1998) [11], Kalembasa (2003) [13], Krzywy & Wołoszyk (1996) [15], Krzywy et al. (1998, 2000, 2002) [16, 17, 19], Krzywy & Jakubowski (2000) [18], and Łabętowicz (2011) [21], which indicate the possible use of sewage sludges for soil conditioning with organic matter and nutrients. The results of microbiological tests of the sewage sludges indicate that the sludges may be used for soil fertilisation and recultivation.

Table 2 provides the quantities of components used for producing the fertiliser mixtures that were then granulated into pellets.

**Table 2**
**Quantities of components used for producing the fertiliser mixtures that were then granulated into pellets**

No. of fertiliser mixture	Recipe for organic and mineral mixture, units				
	Municipal sewage sludge	Industrial sewage sludge	Sawdust from deciduous and coniferous trees	Saturated solution of iron (II) sulphate (VI) 7-hydrate	Saturated solution of urea
	g of dry matter			g of saturated solution	
1	-	-	600	420	180
2	-	600	400	500	500
3	-	600	400	700	300
4	600	-	400	700	300

Table 3 provides chemical characteristics of 4 types of fertilisation pellets, indicating their suitability for fertilisation purposes; included are values of pH<sub>H<sub>2</sub>O</sub>, organic carbon levels and macroelement levels in obtained pellets. Heavy metal levels were not quantified, since they were considerably lower in components used for the production of mixtures than specified by the norms for organic and mineral fertilisers, as set forth in the Regulation of the Minister of Agriculture and Rural Development (Journal of Laws of 2008 No. 119, item 765).

Data provided in Table 3 indicate that:

- the highest organic carbon levels were found in fertilisation pellets produced from municipal sewage sludge, the sawdust mixture, and saturated aqueous solutions of iron (II) sulphate (VI) 7-hydrate and urea; fertilisation pellets produced with industrial sewage sludge, mixture of saturated aqueous solutions of iron (II) sulphate (VI) 7-hydrate, urea and sawdust mixture contained from 182 to 190 g of organic C·kg<sup>-1</sup> of dry matter
- the highest nitrogen levels were observed in fertilisation pellets produced with industrial sewage sludge, mixture of sawdust, saturated aqueous solution of iron (II) sulphate (VI) 7-hydrate and urea. This was caused by the fact that this type of pellets was created using the highest volume of aqueous solution of urea
- the highest total phosphorus, calcium, magnesium and sulphur levels were found in fertilisation pellets produced with municipal sewage sludge; the lowest total phosphorus, potassium, calcium and magnesium levels were found in fertilisation pellets produced from the mixture of sawdust and saturated aqueous solutions of iron (II) sulphate (VI) 7-hydrate and urea
- the highest total sulphur levels were found in fertilisation pellets produced from municipal sewage sludge, sawdust, saturated aqueous solutions of iron (II) sulphate (VI) 7-hydrate and urea, and from industrial sewage sludge, mixture of sawdust and saturated aqueous solutions of iron (II) sulphate (VI) 7-hydrate and urea
- the highest total potassium levels were found in fertilisation pellets produced with industrial sewage sludge.

Table 3

## Chemical properties of fertilisation pellets

Assay or quantification type	Type of organic and mineral pellets. Numerated acc. to Table 2							
	1		2		3		4	
pH <sub>H<sub>2</sub>O</sub>	3,80		5,60		5,20		5,20	
	g·kg <sup>-1</sup> of d.m.	%	g·kg <sup>-1</sup> of d.m.	%	g·kg <sup>-1</sup> of d.m.	%	g·kg <sup>-1</sup> of d.m.	%
Organic C	262	26.2	190	19.0	182	18.2	427	42.7
<b>NIR<sub>0.05</sub> for g·kg<sup>-1</sup> of dry matter 2.27</b>								
Total nitrogen (N)	84.50	8.45	145	14.50	115.2	11.50	134.30	13.40
<b>NIR<sub>0.05</sub> for g·kg<sup>-1</sup> of dry matter 2.45</b>								
Phosphorus (P)	0.25	0.02	4.20	0.42	4.00	0.40	13.05	1.30
<b>NIR<sub>0.05</sub> for g·kg<sup>-1</sup> of dry matter</b>								
Potassium (K)	0.95	0.09	6.20	0.62	6.60	0.66	3.00	0.30
<b>NIR<sub>0.05</sub> for g·kg<sup>-1</sup> of dry matter 0.21</b>								
Calcium (Ca)	0.50	0.05	9.36	0.94	9.38	0.94	13.90	1.39
<b>NIR<sub>0.05</sub> for g·kg<sup>-1</sup> of dry matter 0.19</b>								
Magnesium (Mg)	0.56	0.06	1.14	0.11	1.60	0.16	2.23	0.22
<b>NIR<sub>0.05</sub> for g·kg<sup>-1</sup> of dry matter 0.07</b>								
Sulphur (S)	52.20	5.25	60.00	6.00	90.00	9.00	92.60	9.26
<b>NIR<sub>0.05</sub> for g·kg<sup>-1</sup> of dry matter 0.55</b>								

Therefore, it can be concluded that in terms of chemical properties the produced fertilisation pellets are suitable for soil conditioning with organic matter and nutrients for plants. In terms of organic carbon levels and general nitrogen, phosphorus and potassium levels, fertilisation pellets produced with municipal or industrial sewage sludge comply with the norms applicable to organic and mineral fertilisers. These are set forth in the Regulation of the Minister of Agriculture and Rural Development (Journal of Laws of 2008 No. 119, item 765). According to the Regulation, organic and mineral fertilisers in solid form should contain not less than 20% of organic matter (i.e. 11.8% of organic C), 1 wt. % of total nitrogen, 0.5 wt. % of phosphorus, as converted into P<sub>2</sub>O<sub>5</sub>, i.e. 0.22% P, 1 wt. % of potassium as converted into K<sub>2</sub>O, i.e. 0.83% K. The acceptable levels of heavy metal impurities in those fertilisers are: Cr – 100 mg, Cd – 5.0 mg, Ni – 60 mg, Pb – 140 mg and Hg – 2.0 mg per 1 kg of dry matter of fertiliser. The fertilisers must not contain any ova of intestinal parasites and *Salmonella* bacteria.

In terms of physical properties, the fertilisation pellets had the form of irregular nodules, 1–3 mm in diameter and lighter than standard granulated mineral fertilisers. The pellets' resistance to mechanical damage was average. Further studies on the physical properties of fertilisation pellets should be aimed at ballasting the pellets and improving their resistance to mechanical damage.

## Summary

Industrial waste (industrial sewage sludge from ZCh POLICE S.A., the mixture of deciduous and coniferous sawdust, iron (II) sulphate (VI) 7-hydrate – by-product of titanium dioxide production at ZCh POLICE S.A. – and municipal sewage sludge from the wastewater treatment plant in Stargard Szczeciński) may be used for the production of fertilisation pellets.

The highest levels of nutrients for plants was found in the fertilisation pellets composed of municipal sewage sludge, the mixture of sawdust from deciduous and coniferous trees, and saturated solution of iron (II) sulphate (VI) 7-hydrate and urea. The pellets can be categorised as low-grade nitrogen-sulphuric fertilisers. The pellets contained 13.4% of nitrogen and 9.26% of sulphur. The lowest levels of nutrients for plants were found in the fertilisation pellets composed of the mixture of sawdust from deciduous and coniferous trees, and saturated aqueous solutions: iron (II) sulphate (VI) 7-hydrate and urea. Fertilisation pellets were lighter than standard mineral fertilisers and exhibited average resistance to mechanical damage.

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