Specification and classification of pelletised dried sewage sludge: Identifying its key properties as a renewable material for enabling environmentally non-harmful energy utilisation

Supplementary Materials: Wastewater treatment efficiency at the CWWTPL, pellets preparation, and their assessment as non-hazardous waste.

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1. Evaluation of the Wastewater treatment efficiency at the CWWTPL

1.1. Wastewater sampling and characterisation

At the CWWTPL [1], the wastewater flow at the inlet and outlet is continuously measured with a Venturimeter and an inline flow meter Prosonic S, Endress + Hauser, based on an ultrasound distance meter between the water level and the sensor, according to the accredited methods: i) DIN 19559-1:1983, Measurement of flow of wastewater in open channels and gravity conduits; General information and ii) DIN 19559-2:1983, Measurement of flow of wastewater in open channels and gravity conduits; venturi flumes.

Table S1: Overview of the performed Sampling campaigns.

Sampling campaign, Nº	Year	Date	Inflow, $m^3\ day^{-1}$	Outflow, $m^3\ day^{-1}$
1	2010	The 6th July	86,500	86,600
2		The 12th October	73,700	71,800
3	2011	The 17th May	86,500	86,600
4		The 17th October	73,700	71,800
5	2012	The 27th September	83,100	82,496
6	2013	The 14th May	79,700	79,900
7		The 21st October	59,600	58,400
8	2014	The 25th May	56,900	52,200
9		The 24th September	70,700	70,800
10	2015	The 9th April	62,500	64,500
11		The 27th October	58,312	59,566
12	2016*	The 24th May	65,148	64,882
13		The 16th October	50,194	51,720
14	2017	The 3rd July	66,814	69,950
15		The 5th July	55,252	58,690
16		The 9th July	43,352	46,730
17	2021	The 12th July	60,110	65,080
18	2022	The 7th June	65,000	68,700

***[2].**

At the influent and effluent, daily wastewater samples are automatically sampled time-proportionally using the CSF48 Liquistation, Endress+Hauser. Samples are prepared, cooled, and stored by i) ISO 5667-10:1992, Water quality, Sampling, Part 10: Guidance on sampling of wastewaters, and ii) ISO 5667-3:2012, Water quality, Sampling, Part

3: Preservation and handling of water samples [2]. From 2010 - 2022, additional representative samples were collected in the spring, summer and autumn (Table 1). 24-hourly, time-proportional representative sub-samples were taken at the influent and effluent to determine the removal efficiency of potential toxic metals (PTMs) (As, Cd, Cr, Cu, Hg, Mo, Ni, Pb and Zn), and metals Sb, Co, Mn, Tl and V from the raw wastewater (from Table 2 to Table 8).

The content of the heavy metals (HMs) in the raw wastewater and treated effluent is monitored by standardised physicochemical measurements: i) digestion of the wastewater sample is made according to EN ISO 15587-2:2003, Water quality — Digestion for the determination of selected elements in water — Part 2: Nitric acid digestion, ii) determination of heavy metals (except mercury) is performed with ISO 17294-2 (relevant edition), Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes and iii) mercury is determined with EN ISO 12846:2012, Water quality — Determination of mercury — Method using atomic absorption spectrometry (AAS) with and without enrichment.

Most of the used methods are accredited according to the Technical Standard EN ISO/IEC 17025 (relevant edition), General requirements for the competence of testing and calibration laboratories and performed by authorised contractors.

1.2. The treatment efficiency for heavy metals evaluation/ calculation

$$\text{Treatment efficiency, \% m/m} = [(Q_{inlet} * c_{inlet} - Q_{outlet} * c_{outlet}) / (Q_{inlet} * c_{inlet})] * 100 \quad (1)$$

Q_{inlet}, Q_{outlet}: the inflow of the raw wastewater/ the outflow of the treated wastewater in $m^3\ day^{-1}$

c_{inlet}, c_{outlet}: the concentration of heavy metal in the raw wastewater/ the treated wastewater

Heavy metal concentration for results with values <LOQ or <LOD [3, 4]:

$$c_{inlet}, c_{outlet} = LOQ/2 \text{ or } LOD/2 \quad (2)$$

1.3. The Heavy metals concentration in wastewater and their biological treatment efficiency with the active sludge

Tables 2 to 8 show results with values lower than LOQ and higher than LOD with <. Results with a value lower than the LOD are given by […]. The treatment efficiency could not be assessed in the case of equally low inlet and outlet concentrations.

Table S2. Overview of the removal efficiency for arsenic and cadmium.

Sampling campaign, Nº	As			Cd		
	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m
1	[0.007]	[0.007]	n. e.	[0.0003]	[0.0003]	n. e.
2	[0.007]	[0.007]		[0.0003]	[0.0003]	
3	[0.007]	[0.007]		<0.0010	[0.0003]	70.0
4	[0.007]	[0.007]		[0.003]	[0.0003]	n. e.
6	[0.007]	[0.007]		[0.0003]	[0.0003]	
7	[0.007]	<0.010	0	[0.0003]	[0.0003]	
8	0.0005	[0.0003]	72.5	[0.0003]	[0.0003]	
9	0.00076	0.00048	36.8	[0.0003]	[0.0003]	
10	0.0010	0.0037	0	[0.0003]	[0.0003]	
11	0.00080	0.00043	45.1	[0.0003]	[0.0003]	
12*	0.00093	0.00046	50.7	[0.0003]	[0.0003]	
13*	0.0018	0.00043	75.4	0.00068	[0.0003]	77.3
17	0.0011	0.00077	24.2	[0.0003]	[0.0003]	n. e.
18	0.00093	0.00061	30.7	0.00019	[0.0003]	16.6

*** [2].**

In general, the content of heavy metals in municipal wastewater is low, most often at the limit of quantification. The methods for sample decomposition and determination of the heavy metals and mercury improve all the time. That resulted in the results' quality and lowered the LOQ and LOD. After 2014 (since campaign 8), the quality of results has improved, and fewer results are as <LOQ or [LOD]. The latter is especially true for As, Sb, Co, and Mn. The Cd, Sb, and Tl concentrations are mostly as [LOD]. Mercury concentration at the outlet of the CWWTPL is on the level of ng L^{-1} or as [LOD].

Table S3. Overview of the removal efficiency for antimony and cobalt.

Sampling campaign, Nº	Sb			Co		
	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m
6	[0.005]	[0.005]	n. e.	[0.003]	[0.003]	n. e.
7	[0.005]	[0.005]	n. e.	[0.003]	[0.003]	n. e.
8	[0.001]	[0.001]	n. e.	0.00096	0.00071	32.2
9	[0.001]	[0.001]	n. e.	0.0016	0.00073	54.3
10	[0.001]	[0.001]	n. e.	0.0022	0.0040	0
11	[0.001]	[0.001]	n. e.	0.0015	0.00033	77.5
12*	[0.001]	[0.001]	n. e.	0.0024	0.00059	75.5
13*	0.0021	[0.001]	75.5	0.0048	0.00053	88.6
14	no a.			0.0015	<0.0005	82.6
15				0.0014	<0.0005	81.0
16				0.00094	<0.0005	71.3
17	[0.001]	[0.001]	n. e.	0.00085	0.00033	58.0
18	<LOD=0.00035	<LOD=0.00037	0	0.0029	0.0005	81.8

*** [2].**

Table S4. Overview of the removal efficiency for chromium and copper.

Sampling campaign, Nº	Cr			Cu		
	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m
1	0.014	0.012	14.2	0.009	<0.005	72.2
2	<0.010	<0.010	n. e.	0.026	<0.005	90.4
3	0.014	0.011	21.3	0.034	<0.0050	92.8
4	0.032	[0.003]	95.4	0.039	0.0065	83.7
5	0.027	0.027	0.73	0.041	<0.0050	87.8
6	0.016	<0.010	68.7	0.029	0.0052	82.5
7	0.011	[0.003]	86.6	0.033	<0.0050	92.4
8	0.011	0.0056	53.3	0.065	0.0091	86.4
9	0.039	0.0088	96.1	0.022	<0.0050	88.7
10	0.041	[0.005]	93.7	0.041	0.0069	83.1
11	0.021	[0.005]	87.8	0.30	0.024	92.2
12*	0.026	[0.005]	90.4	0.012	[0.01]	61.8
13*	0.049	[0.005]	94.7	0.061	[0.01]	91.8
17	0.0068	0.0015	76.1	0.068	[0.01]	92.4
18	0.019	[0.005]	86.1	0.015	[0.01]	64.8

*** [2].**

Table S5. Overview of the removal efficiency for thallium and vanadium.

Sampling campaign, Nº	Tl			V		
	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m
6	[0.00025]	[0.00025]	n. e.	[0.030]	[0.030]	n. e.
7	[0.00025]	[0.00025]		[0.030]	[0.030]	
8	<0.01	<0.001	90.8	0.0014	0.00081	46.9
9	[0.005]	[0.005]	n. e.	0.0016	0.00056	65.0
10	[0.005]	[0.005]		0.0075	0.00034	95.3
11	[0.005]	[0.005]		0.0025	0.00043	82.4
12*	[0.005]	[0.005]		0.0030	0.00055	81.7
13*	[0.005]	[0.005]		0.0075	0.00032	95.6
17	[0.005]	[0.005]		0.0028	0.00066	74.5
18	[0.005]	[0.005]		0.0031	0.00059	79.9

*** [2].**

Table S6. Overview of the removal efficiency for mercury and zinc.

Sampling campaign, Nº	Hg			Zn		
	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, % m/m
1	[0.00015]	[0.00015]	n. e.	0.29	<0.10	65.5
2	<0.00020	[0.00015]	26.9	0.20	<0.10	75.6
3	<0.00020	[0.00015]	24.9	0.26	<0.10	80.7
4	0.00065	[0.00015]	88.8	0.31	<0.10	84.3
5	[0.00015]	[0.00015]	n. e.	0.14	<0.10	64.5
6	0.00025	[0.00015]	69.9	0.19	<0.10	73.6
7	0.00079	[0.00015]	90.7	0.35	<0.10	86.0
8	0.00032	[0.00003]	95.7	0.21	0.098	57.2
9	0.00014	[0.00003]	89.3	0.18	[0.05]	86.1

10	0.00019	0.000093	49.5	0.25	0.087	64.1
11*	0.00013	[0.000007]	97.2	0.20	0.074	62.2
12*	0.00011	0.000016	85.5	0.24	0.10	58.5
13	0.0080	0.000042	99.5	0.61	0.065	89.0
17	no a.	[0.000007]	n. e.	0.12	0.047	57.6
18	0.00019	0.000039	78.3	0.28	0.065	75.5

* **[2].**

Table S7. Overview of the removal efficiency for nickel and lead.

Sampling campaign, Nº	Ni			Pb		
	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, %	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, %
1	0.011	<0.010	54.5	0.031	[0.0030]	95.2
2	<0.010	<0.010	n. e.	0.010	[0.0030]	85.4
3	0.015	<0.010	66.6	0.0097	[0.0030]	84.5
4	0.028	0.017	40.9	0.016	[0.0030]	90.9
5	0.049	0.014	71.6	0.0078	[0.0030]	80.9
6	0.015	0.010	33.2	0.015	[0.0030]	90.0
7	0.063	<0.010	92.2	0.029	[0.0030]	94.9
8	0.0073	0.0079	0.72	0.010	[0.003]	86.2
9	0.018	0.0075	58.3	0.018	[0.003]	91.7
10	0.044	0.027	36.7	0.016	[0.003]	90.3
11	0.011	0.0058	46.1	0.010	<0.005	74.5
12*	0.021	0.0056	73.4	0.018	[0.003]	91.7
13*	0.033	0.0067	79.1	0.060	[0.003]	97.4
17	0.01	0.0066	28.5	0.006	[0.003]	70.5
18	0.017	0.0074	54.0	0.024	[0.003]	93.4

*** [2].**

Table S8. Overview of the removal efficiency for manganese and molybdenum.

Sampling campaign, Nº	Mn			Mo		
	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, %	Inlet concentration, mg L^{-1}	Outlet concentration, mg L^{-1}	Treatment efficiency, %
6	<0.10	[0.025]	74.9	no a.		
7	<0.10	[0.025]	75.5			
8	0.043	0.017	63.7			
9	0.054	0.017	68.5			
10	0.064	0.029	53.2			
11	0.058	0.0069	87.8			
12*	0.057	0.015	73.8			
13*	0.11	0.011	89.7			
17	0.041	0.0083	78.1	0.0018	0.00091	45.3
18	0.25	0.029	87.7	0.00054	0.00024	53.0

*** [2].**

2. Pellets production at the CWWTPL [1]

The entire CWWTPL quality control system has been set up according to ISO 9001:2015 - Quality management systems, Requirements. To achieve the objectives of the special national demands on solid recovered fuel (SRF) production, the existing quality management system must be upgraded with specific demands for the operation of facilities for the production and trade of SRF [5 - 7], covering operations from the point of acceptance of surplus sludge destined for recovery to the point of delivery of the SRF shipment to the final stakeholder in accordance with the contract (Figure 1).

Raw surplus sludge is discharged daily from the biological system. After gravitational and mechanical pre-thickening with the addition of flocculants, the excess sludge contains 5.5 to 6.5 % of dry matter. It is then alternately taken to one of two identical parallel digesters capable of holding a total volume of 14,800 m^3 and processed through anaerobic mesophilic digestion. The digested sludge contains 3.0 - 3.5 % dry matter. It is collected into a 1,850 m^3 secondary thickening tank. When the tank is full, the digestate dehydration process starts. Afterwards, it is transported to the mixer, where dry granulates are added. This mixture reaches a moisture content of 55 – 65 % dry matter. In this rheological form, it is suitable to be exposed to thermal treatment in a convectional rotating drying drum (Figure 2). The latter process is not continual but is in batch mode connected with the running of the centrifuge. Drum performs mixture drying up to 92 % m/m of dry matter, pelleting, and hygienisation simultaneously. Final pellets have 2 mm to 4 mm in diameter. Until taken over by the stakeholder, pellets are temporarily stored in a silo (Figure 2) with a volume of 50 m^3.

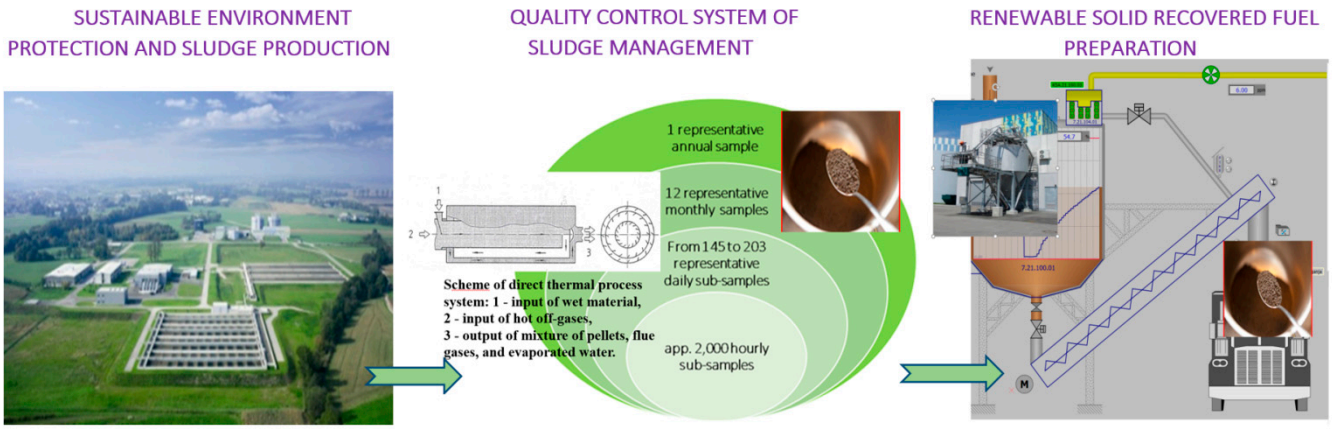

Figure S1. Sewage sludge management at the CWWTPL [1, 2, 5 - 7].

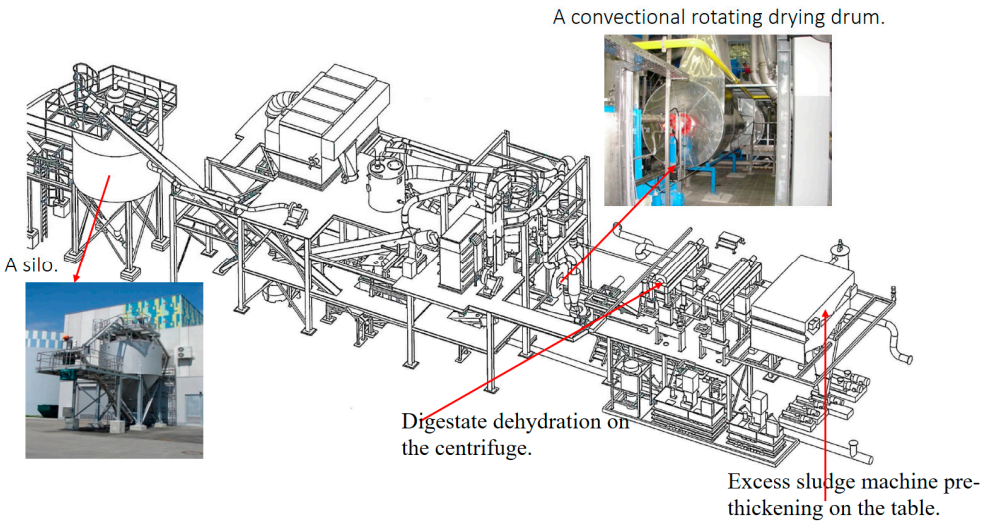

Figure S2. Schematic representation of mechanical pre-treatment of excess sludge and digestate at the CWWTPL [8–11].

3. The pellets assessment according to hazardous waste characteristics

Hazardous waste« means waste which displays one or more of the hazardous properties listed in Annex III of Directive 2008/98/EC [12]. Based on the review of pellets generation technology at the Central wastewater treatment plant, the review of input raw materials, pellets composition, and assessment of analysis results on the annual representative samples, the report was prepared regarding the content of hazardous matter.

Official reports NLZOH [13] and NLZOH [14] state that the waste in question does not contain substances that could be classified under one of the hazard statements [15], and designated as additional hazardous items related to HP 15 [12, 15, 16]. 'Hazard statement' means a phrase assigned to a hazard class and category that describes the nature of the hazards of a hazardous substance or mixture, including, where appropriate, the degree of risk. By the EU Commission Regulation [16], the hazardous properties H 1 to H 15 defined in Annex III to Directive 2008/98/EC [12] are renamed to HP 1 to HP 15 to avoid potential confusion with the hazard statement codes defined in EC Regulation [15] (Table 9). Directive 2008/98/EC [12] states that the classification of waste as hazardous should be based, among other things, on the EU Union legislation on chemicals, in particular concerning the type of preparations as hazardous, including concentration limit values used for that purpose.

Table S9. The evaluation of pellets according to hazardous waste characteristics [12 - 14].

Properties of waste which render it hazardous	Description	Assessment report (Yes/No)
HP 1	**"Explosive":** waste is capable of chemical reaction of producing gas at such a temperature and pressure and at such a speed as to cause damage to the surroundings.	No
HP 2	**"Oxidising":** waste, which may, generally by providing oxygen, cause or contribute to the combustion of other materials.	No
HP 3	**"Flammable":** flammable solid waste, which is readily combustible or may cause or contribute to fire through friction.	No
HP 4	**"Irritant — skin irritation and eye damage"** waste on the application can cause skin irritation or damage to the eye.	No
HP 5	**"Specific Target Organ Toxicity (STOT)/Aspiration Toxicity"** can cause specific target organ toxicity either from a single or repeated exposure or cause acute toxic effects following aspiration.	No
HP 6	**"Acute Toxicity"** can cause acute toxic effects following oral or dermal contact or inhalation exposure.	No
HP 7	**"Carcinogenic":** waste, which induces cancer or increases its incidence.	No
HP 8	**"Corrosive":** waste, which on the application can cause skin corrosion.	No
HP 9	**"Infectious":** waste containing viable micro-organisms or toxins reliably believed to cause disease in man or other living organisms.	No
HP 10	**"Toxic for reproduction":** waste affects sexual function and fertility in adult males and females, as well as developmental toxicity in the offspring.	No
HP 11	**"Mutagenic":** waste may cause a mutation, a permanent change in the amount or structure of the genetic material in a cell.	No
HP 12	**"Release of an acute toxic gas":** waste releases acute toxic gases in contact with water or acid.	No
HP 13	**"Sensitising":** waste contains one or more substances known to cause sensitising effects to the skin or the respiratory organs.	No
HP 14	**"Ecotoxic":** waste presents or may present immediate or delayed risks for one or more sectors of the environment.	No
HP 15	**"Waste capable of exhibiting a hazardous property listed above not directly displayed by the original waste".**	No

4. The SRF classification into Quality classes

The Technical Report [17] summarises a classification system, classes, and specification procedure to evaluate the treated waste as an SRF by the decision of CEN/TC 343 Working Group 2, Fuel Specification and Classes. Technical Report [17] introduced a list of three operational parameters essential for the successful operation of a combustion facility: i) economics aspects (NCV), ii) corrosion (Cl) and emission indicators(Hg and Cd). Cadmium is added as a proposal to supplement the classification system, which can be used if needed. The classification system presents five classes (Table 10). At least ten representative samples of treated waste, sampled evenly over a calendar year, must be analysed to specify the Class code. The highest class (the worst SRF Quality class) is reserved for the SRF derived from, e.g. sewage sludge and filter cakes. Technical Standard [6], superseded with Technical Standard [7] in May 2021, determines the quality classes based only on NCV, Cl, and Hg. Both documents determine the method of evaluating the energy and environmental suitability of treated waste for use as SRF and set the statistical procedures for generated analytical results.

Table S10. Classification of SRFs [17].

Classification characteristic	Statistical measure	Unit	*Classes*[a]				
			1	2	3	4	5
NCV	Arithmetic mean	$MJ \cdot kg^{-1}_{ar}$	*≥ 25*	≥ 20	≥ 15	≥ 10	≥ 3
Chlorine (Cl)		% m/m_{DM}	*≤ 0.2*	≤ 0.6	≤ 1.0	≤ 1.5	≤ 3
Mercury (Hg)	Median	$mg \cdot MJ^{-1}_{ar}$	*≤ 0.02*	≤ 0.03	≤ 0.08	≤ 0.15	≤ 0.50
	80th percentile		*≤ 0.04*	≤ 0.06	≤ 0.16	≤ 0.30	≤ 1.00
Cadmium[b] (Cd)	Median		*≤ 0.1*	≤ 0.30	≤ 1.0	≤ 5.0	≤ 15[c]
	80th percentile		*≤ 0.2*	≤ 0.60	≤ 2.0	≤ 10	≤ 30[c]

[a] Values refer to a minimum of 10 analyses. [b]Proposed classes for Cd. [c]For the SRF with high ash content and therefore a higher raw material substitution in the clinker production with a maximum of 100 mg kg^{-1}_{ar}.

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