

Pyrolysis - the initial stage of an integrated approach to optimal material recovery of sewage sludge

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Abstract

Dried sewage sludge is a non-hazardous waste in the shape of pellets with the diameter from 2 mm to 4 mm. Pellets consist of high proportion of organic matter and have low moisture content. Its final treatment must be focussed in the optimum energy-efficient conversion of organic part and material utilization of nutrients (phosphorus, potassium and nitrogen). Optimum recovery of these substances can be achieved by pyrolysis. This process of thermochemical conversion of pellets was carried out to produce four phases: i) a solid phase (biochar), ii) the heavy liquid phase - pyrolysis oil, iii) a light liquid fraction – aqueous phase and iv) gaseous phase. Such pre-treatment of dried sewage sludge and further processing of products formed during the pyrolysis may change their status from waste to new product.

Key words: biochar, dried sewage sludge, extra light fuel oil, nutrient recovery, pyrolysis, reaction water.

INTRODUCTION

Anaerobically stabilized excess sludge (further referred to as raw digestate) is a source of organic carbon and nutrients. Because of high proportion of water amount, raw digestate must be mechanically dehydrated to reduce excess water. A reject water stream (further referred to as centrate) contains a high concentration of ammonia nitrogen and orthophosphate, and therefore it is an important potential source of these nutrients. The organic compounds present in centrate are predominantly poorly biodegradable, as evidenced by the ratio of COD:BOD₅, which is usually 10:1. Mechanically thickened digestate in the form of a cake contains about 80% of the centrate, thus, also substances that are present in centrate.

At WWTP (website 2016, b), the conventional pre-treatment process of surplus biomass, including dehydrated raw digestate, is followed in a convectional rotating drying drum. The pelletized sewage sludge (further referred to as pellets) has 91 % - 93 % of dry matter content. Pellets are 2 mm – 4 mm in size and have a bulk density of 650 kgm⁻³ - 700 kgm⁻³. They still contain a high proportion of organic matter and are utilised as an additional alternative solid recovered fuel (website 2016, a). The composition of dried sewage sludge is very different from the centrate. It consists mainly of biomass or its macromolecules, intermediate anaerobic degradation products of surplus sludge, the inert inorganic substances and inorganic substances as a result of the degradation of biomass.

Drying of the sludge and the pelleting is carried out at T = 92 °C, and for this reason struvite (magnesium ammonium phosphate, NH₄MgPO₄·6H₂O), present in the raw digestate, decomposed, so that its final content in the pellets is only present to a small extent. Still, the pellets contain

orthophosphate, ammonium nitrogen and organically bound nitrogen. Also, pelletized sludge contains sufficient organic carbon, which in addition to the appropriate low moisture content, have an adequate calorific value. Because a sufficient content of organic share and the relatively high proportion of ash, which has a complex composition, it is necessary to think about the overall utilization of the pellets, i.e. energy and material utilization.

Holistic approach to final material and/or energy utilization of sewage sludge still represents a major challenge for R&D in the processing of sewage sludge (Fytili and Zabaniotou 2008). Its final treatment must be focussed in the optimum energy-efficient conversion of organic part, material utilization of nutrients such as phosphorus, potassium and nitrogen, and a practical final recovery of inorganic residues resulting from energy utilization of sludge. Optimum recovery of these substances can be achieved by pyrolysis of sewage sludge through pre-treatment process followed by final processing.

Pyrolysis is a thermochemical decomposition of biomass into a range of useful products. This process is typically carried out in a temperature range from 300 °C to 650 °C. During pyrolysis, large complex hydrocarbon molecules of biomass break down into relatively smaller and simpler molecules of gas, liquid, and biochar (Prabir 2013; Dahlquist 2013).

The resulting volatile pyrolysis products are fractionated and isolated into separate groups of substances with different chemical properties. Solid fraction (further referred to as biochar) contains organic and inorganic part. Gaseous phase leaves the reactor system, and the liquid phase is condensed into a light oily fraction and a heavy fraction (further referred to as pyrolysis oil). Most water resulting from the moisture, chemically or crystalline bounded water and water generated during the thermal decomposition of the substance, is collected in the light oily fraction (further referred to as aqueous phase) and contains a high concentration of condensed water-soluble substances (Mislej et al 2012).

MATERIALS AND METHODS

Materials

The laboratory prepared centrate was prepared by centrifuging the raw digestate without the addition of polyelectrolyte in a laboratory centrifuge (Tehtnica, CENTRIC 322A, at speed 4800 r.p.m.). Thickened sludge (further referred to as digestate) and centrate were separated by decantation, resulting centrate (further referred to as centrate) was then filtered through the filter Sartorius, Minisart 17594-K. To determine the phases by XRD analysis and qualitative elemental analysis, both fractions of raw digestate were dried in a laboratory drying oven at $T = 40$ °C. Comparatively, we also analyzed the centrate produced at full scale (further referred to as centrate_p) by performing the regular treatment of raw digestate (centrifuge dehydration with addition of cationic polyelectrolyte). Additionally to analysis of centrate, we also analyzed two samples of pellets. The sampling was performed in the time of the regular process of the excess sludge treatment during the whole production year. We have prepared two annual composed representative samples of pellets, one for the year 2012 (pellets 2012) and another one for the year 2014 (pellets 2014).

Semi-pilot pyrolysis reactor

On the lab scale, the pyrolysis of granulated dried sewage sludge (without further grinding) was performed (Picture 1) in the semi-pilot batch reactor.

This small reactor of cylindrical shape, with 10 cm diameter, 21.5 cm height and a volume of 1.69 L (Picture 1) was used for pellets pyrolysis in an amount from 400 g to 500 g. The heating rate was 12 Kmin⁻¹ and the maximum temperature of the outer layer was 600 °C. The process was performed without purging and with an absolute pressure of 800 mBar.

Prior to pyrolysis, pellets were dried in a laboratory oven at 105 °C to achieve nearly complete dryness. The pyrolysis was carried out to produce four phases: i) a solid phase (biochar), ii)

pyrolysis oil, iii) aqueous phase and iv) gaseous phase. The last one was transported to Bunsen burner figure as a flare. The amount of produced non-condensable gases was measured with a gas flow meter. The temperature inside the reactor was in the temperature range from 450 °C to 520 °C. The generated volatiles were separately recovered by fractionation. We performed two type of fractionation of volatiles. By first procedure (experiment A) we had got only one liquid phase (pyrolysis oil 2012 and aqueous phase) and gasous phase. With the second procedure (experiment B) we generated two type of liquid fractions - the first heavy oily fraction (pyrolysis oil 2014) was condensed at a temperature from 120 °C to 135 °C, followed by cooling of volatile compounds (aqueous phase 2014) with a water cooler. The non-volatile part of pellets was retained in the reactor as biochar. The resulting products of experiment A and sample of pellets marked as pellets 2012 were: i) biochar BC 2012 and ii) pyrolysis oil 2012, and the resulting products of experiment B and sample pellets 2014 were: i) biochar BC 2014, ii) pyrolysis oil 2014 and iii) water phase 2014.



Figure 1: Semi-pilot pyrolysis reactor

Analytical Methods

Standardized methods were used for physicochemical analysis of pellets (Grilc et al 2010), biochar and pyrolysis oil. Additionally, commercial test kits were used for chemical analysis of centrate and aqueous phase (Hach lange Test kits).

Mineralogical characterization of the resulting solid residue (biochar) has been accomplished by a combination of two methods: i) morphology, microstructure and semi-quantitative chemical analysis by scanning electron microscopy (SEM) and electronic dispersion spectroscopy (EDS) with the use of the apparatus JEOL 5500 LV and ii) x-ray diffraction (XRD), which was carried out by powder X-ray diffraction in the X-ray powder diffractometer Empyrean, PANalytical; analysis were conducted at voltage of 45 kV and 40 mA current with a Cu K α anode, in an angular range of 20-70 °(2 θ) with 0.02 °(2 θ) step and integration time of 200 s.

Table 3. Methods used for physico-chemical analysis of pyrolysis oil

Parameter	Methods
Density	EN ISO 12185:1996
Kinematic Viscosity at 40 °C	ASTM D 7042
Determination of carbon residue	EN ISO 10370:2014
Corrosiveness to copper, (3h at T=50 °C)	EN ISO 2160:1998
Flash point determination), PM	EN ISO 2719:2002
Determination of pour point	ISO 3016:1994
Water content	ASTM D 6304

Quality of resulting pyrolysis oil was examined with the methods prescribed in Slovenian Technical

Standard BS 1011 (Table 3) regarding the quality of extra light fuel oil(Standardization 2011).

Materials characterization

The SEM/EDS analysis of the centrate is shown in Fig. 2, where three different phases can be seen: i) the first is composed of P and K, ii) the second from K and Cl (mineral phase sylvite) and iii) the third from Mg and P (mineral phase struvite).

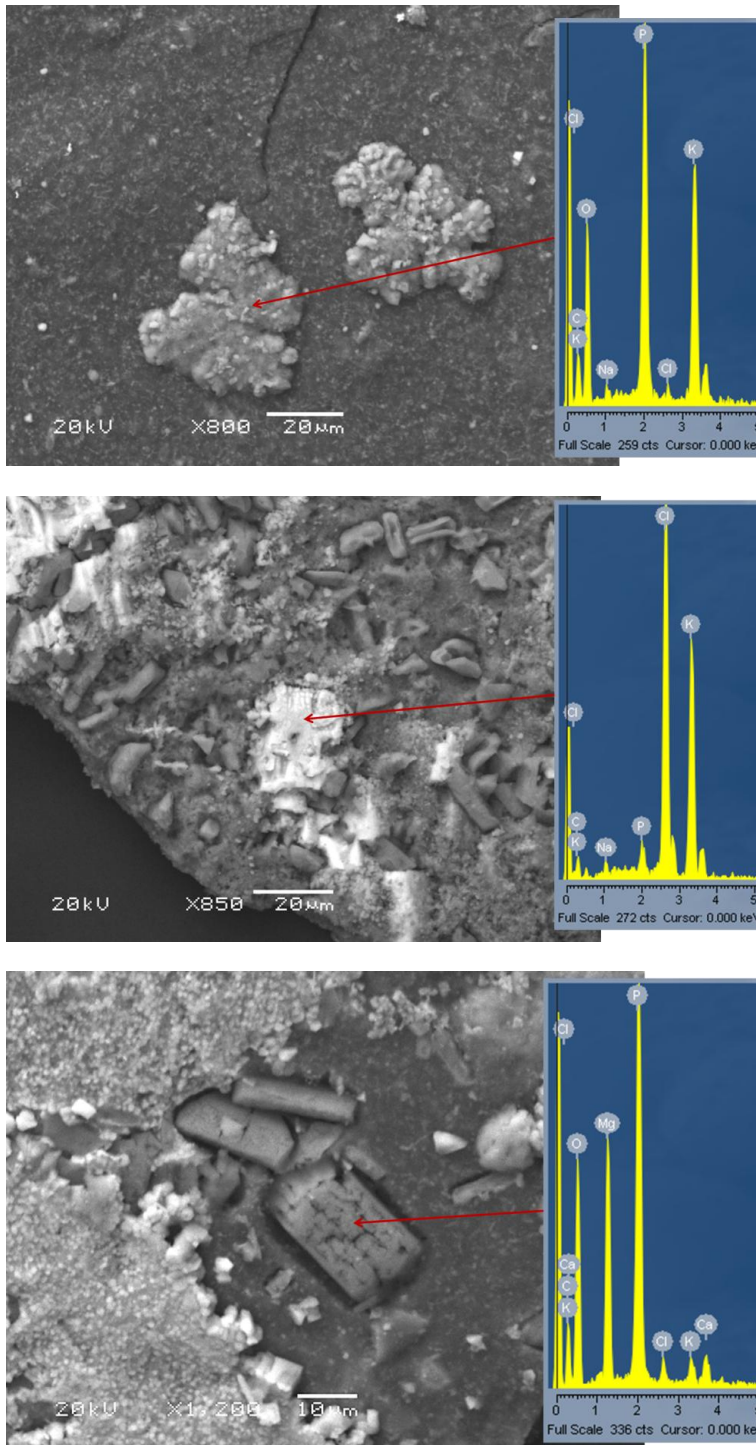


Figure 2. SEM/EDS analysis of the centrate.

Table 1. Physico-chemical analysis of centrate

Parameters	Methods	Units	Centrate	Centrate _p
pH	ISO 10523:2008		8.3	7.9
Alkalinity	ASTM D 1067-B _{mod}	mg CaCO ₃ /L	6,050	6,095
Electrical Conductivity (25 °C)	EN 27888:1993	mS/cm	12.6	12.8
VFA	Hach Test Kit, LCK 365 SM 4500-NH ₃ B.;	mg CH ₃ COOH/L	339	109
NH ₄ -N	SM 4500-NH ₃ C.; Hach Test Kit, LCK 303	mg/L	1,781	1,780
N _{tot}	DIN 38409-H28:1992	mg/L	1,812	1,886
COD	ISO 15705:2002	mg O ₂ /L	2,013	430
BOD ₅	ISO 5815-1:2003	mg O ₂ /L	135	55
PO ₄ -P	EN ISO 10304-1:2007; Hach Test Kit, LCK 348	mg/L	114	178
Dry residue at T=105 °C	SM 2540 B, 22 nd Ed.	mg/L	2,960	2,940
Ash at T=550 °C	SM 2540 B, 22 nd Ed.	mg/L	1,180	860

To facilitate understanding of the thermochemical conversion and decomposition of dried sludge, we performed physico-chemical analysis of centrate (Table 1), thickened raw digestate and pellets (Table 2).

Table 2. Physico-chemical analysis of row pellets

Parameters	Methods	Units	Pellets 2012	Pellets 2014
pH	ISO 10523	-	7,8	
Electroconductivity (25 °C)	EN 27888	mS/m	238	
Bulk density	EN 12580	kg/m ³	647	683
Specific surface area, BET		m ² /g	0.915	0.915
Dry matter	EN 14346	%	91.1	91.5
Ash at T= 550 °C	SM 2540 G, 22 nd Ed.	%	29.9	29.9
Ash at T= 900 °C	EN 15402:2011	%	26.0	27.0
Volatile matter at 900 °C	EN 15402:2011	% w/w DS	53.9	53.3
TOC	EN 13137, Method B	C, % w/w DS	38.4	39.5
TC		C, % w/w DS	40.4	39.5
Total Nitrogen	EN 13654	N, % w/w DS	6.15	5.58
NH ₄ -N	ISO 5664	N, % w/w DS	1.0	n.a.
Total Phosphorus	ISO 6878	P, % w/w DS	2.2	1.77
Organic matter	EN 13039	% w/w DS	66.9	n.a.
Mercury	EN ISO 12846	Hg, mg/kg DS	1.8	2.3
Σ Heavy metals*	TS CEN/TS 15411	mg/kg DS	1,000	1,140
Upper calorific value		MJ/kg	14.319	14.09
Net calorific value	EN 15400	MJ/kg	13.281	13.20
Sulphur	EN 15408	S, % (w/w) DS	1.15	1.26
Hydrogen	ISO 609	H, % (w/w) DS	4.4	5.08

*(Sb, As, Cr, Co, Cu, Pb, Mn, Ni, V)

RESULTS AND DISCUSSION

Pyrolysis products

The weight percent of the fractions were as follows (experiment A): i) a 50.3% w/w_{DS} biochar, ii) 16.8% w/w_{DS} liquid oil phase, iii) a 15.1% w/w_{DS} of the gas phase respectively 50.6 normal L of combustible gas mixture, and iv) a 17.8% w/w_{DS} of the aqueous liquid phase.

The main characteristics of biochars and pyrolysis oil are presented in Table 4.

Table 4. Physico-chemical analysis of pyrolysis products

Parameters	Methods	Units	BC 2012	BC 2014	Pyrolysis oil 2012
pH	ISO 10523	-	11		
Electrical Conductivity (25 °C)	EN 27888	mS/m	48		n.a.
Bulk density	EN 12580	kg/m ³	608		
Specific surface area, BET		m ² /g	6.33		
Dry matter	EN 14346	%	99.5	100.0	80.8
Ash at T= 550 °C	SM 2540 G, 22 nd Ed.	%		60.1	
Ash at T= 900 °C	EN 15402:2011	%		54.8	
Volatile matter at 900 °C	EN 15402:2011	% w/w DS		43.9	
TOC	EN 13137,	C, % w/w DS	32.4		91.4
TC	Method B	C, % w/w DS	32.1		91.4
Total Nitrogen	EN 13654	N, % w/w DS	3.57		8.3
NH ₄ -N	ISO 5664	N, % w/w DS		n.a.	
Total Phosphorus	ISO 6878	P, % w/w DS	4.13		
Organic matter	EN 13039	% w/w DS	37.1		
Mercury	EN ISO 12846	Hg, mg/kg DS	0.082		1.7
Σ Heavy metals	TS CEN/TS 15411	mg/kg DS	1,600		56
Upper calorific value		MJ/kg	11.22		32.831
Net calorific value	EN 15400	MJ/kg	11.07		30.978
Sulphur	EN 15408	S, % (w/w) DS	0.38		1.03
Hydrogen	ISO 609	H, % (w/w) DS	1.06		9.42

Calorific value of the oil phase and its hydrogen content were found relatively high (Table 4) and as such, the pyrolysis oil product seems fairly promising, but its high mercury content may imply serious ecological limitations. It was found that resulting pyrolysis oil is not suitable for use in conventional heating systems in its crude form because of insufficient calorific value, for which the required value is above 42.6 MJkg⁻¹ (Standardization 2011). There are also other quality parameters of the pyrolysis oil which deviate from the prescribed values for relevant quality of light fuel oil (Table 5), such as: i) kinematic viscosity, ii) carbon residue, iii) flash point, iv) pour point of petroleum products and v) water content (Standardization 2011).

We could predict that the pyrolysis oil (Pyrolysis oil 2014), which was isolated with the implemented procedure (experiment B), is suitable only as alternative liquid fuel in industrial furnaces.

Table 5. Quality characteristics of pyrolysis oil as a fuel oil extra light

Parameters	Units	Limit value	Pyrolysis oil 2014
Density	kgm ⁻³	< 860	915.4
Kinematic Viscosity at 40 °C	mm ² s ⁻¹	2.5 - 6.0	57.08
Determination of carbon residue	%		
Corrosiveness to copper, (3h at T=50 °C)	(m/m)	≤ 0.15	4.45
Flash point determination), PM	-	1b	/
Determination of pour point	°C	> 55	37.5
Water content	°C	-9	12
	mgkg ⁻¹	≤ 200	31,970

Biochar

Biochar is by mass the major pyrolytic product. It shows potential characteristics for application in agriculture due to its high immobilization affinity of heavy metals (Mislej et al 2015, b). In addition, biochar contains a higher content of phosphorus and nitrogen (Table 4), which further demonstrates the great potential of its use as a fertilizer.

Table 6. Test Category A - Basic Utility Properties of biochar

Parameters	Units	Test Category A – Basic Utility Properties	BC 2014
pH	-		11.0
Electrical Conductivity (25 °C)	mS/m		48
Moisture	%	DECLARATION	0.5
Ash at T = 550 °C	% m/m _{s.s.}		62.3
Ash at T = 900 °C	% m/m _{s.s.}		56.3
TOC	C, % m/m _{s.s.}	10% Minimum Class 1: ≥60% Class 2: ≥30% and <60% Class 3: ≥10% and <30%	32.4
H : TOC	Molar ratio	0.7; Maximum	5.09
Total Nitrogen	N, % m/m _{s.s.}	DECLARATION	3.57
Liming (if pH is above 7)	CaCO ₃ , % m/m _{s.s.}		22.1

As regards compliance with the technical requirements for biochar "Standardized Product Definition and Product Testing Guidelines for Biochar That Is Used in Soil" (Standardization 2014) it is estimated, that the resulting biochar has the following quality (Table 6): i) CLASS 2 according to the "Test Category A - Basic Utility Properties" with remark that the molar ratio H: TOC indicates the carbon instability, ii) in relation to the "Test Category B - toxicant assessment", it is estimated that biochar does not show toxicity (Table 7), but with remark that a significant variations in the international toxicant regulations and thresholds used for determining the range of maximum allowed metals content for biochar materials (Standardization 2014).

Table 7. Test Category B - toxicant assessment of biochar

Parameters	Units	Test Category B – Maximum Allowed Thresholds	BC 2012
Arsenic	As, mg/kg d.s.	13 - 100	4.2
Cadmium	Cd, mg/kg d.s.	1.4 - 39	n.a.
Chromium, total	Cr, mg/kg d.s.	93 - 1200	29
Cobalt	Co, mg/kg d.s.	34 - 100	32
Copper	Cu, mg/kg d.s.	134 - 6000	610
Lead	Pb, mg/kg d.s.	121 - 300	160
Mercury	Hg, mg/kg d.s.	1 - 17	0.082
Molybdenum	Mo, mg/kg d.s.	5 - 75	n.a.
Nickel	Ni, mg/kg d.s.	47 - 420	150
Selenium	Se, mg/kg d.s.	2 - 200	
Zinc	Zn, mg/kg d.s.	416 - 7400	
Sodium	Na, mg/kg d.s.		n.a.
Boron	B, mg/kg d.s.	DECLARATION	
Chlorine	Cl, mg/kg d.s.		

The results of SEM/EDS analysis showed that the majority of fragments of solid phases (pellets and biochar) consists of Si, Ca, Mg, C and O, or a combination of those elements. Fragments consisting of Mg and P can also be relatively frequent detected. Phosphorus appears in combination with Mg and Ca, while the K and Na are detected in combination with Si, Al, Ca and Fe, while sulphur is found in combination with Ca, and also with Fe (Mislej et al 2015, a,b).

Further, the XRD analysis showed that the silica, calcite and dolomite are the most common mineral phases in the investigated samples. X- ray diffraction analysis (Fig. 3) revealed the crystalline structure and composition of minerals present in the investigated samples, with quartz (SiO₂), calcite (CaCO₃) and dolomite (CaMg(CO₃)₂) detected as mayor phases in the digestate, pellets 2014 and BC 2014. The phase composition of the centrate differs from all of the other samples, since sylvite (KCl), archerite (KH₂PO₄) and/ or its ammonium analogue biphosphammite ((K, NH₄) H₂PO₄) are represented as prevailing phases. In the centrate, struvite ((NH₄) Mg (PO₄) 6H₂O) and apthitalite (K, Na)₃ Na (SO₄)₂ were also defined. The struvite was also detected in the digestate and pellets 2014, but not in BC 2014, as due to the high temperatures, it decomposed by dehydration and transformation. Broad hump in the range of 20 – 25 °(2θ) observed in the digestate and pellets 2014 reflects the amorphous (low crystalline) structure of biomass feedstocks. The decrease in the intensity of this broad hump after the pyrolysis was observed in BC 2014. It indicates destruction of biomass structure during the pyrolysis.

Because of other favourable physical properties of the biochar, this pyrolysis product could be utilized as activated charcoal after appropriate activation procedure is performed (Zagar 2014).

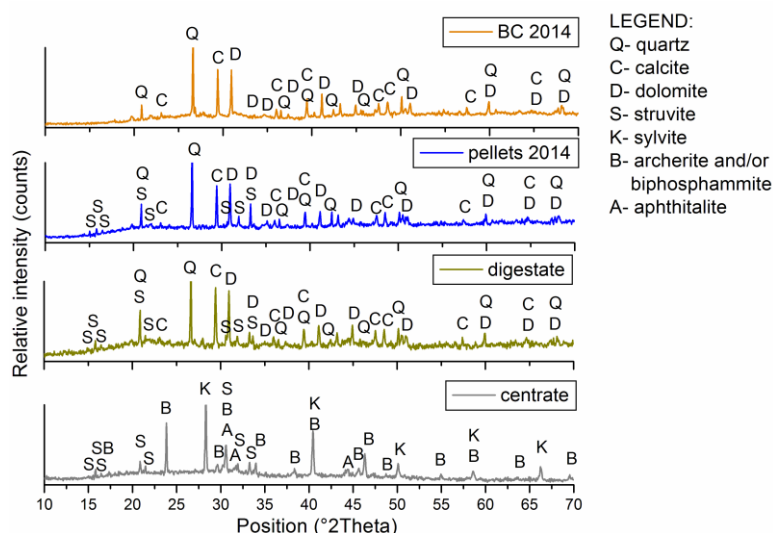


Figure 3. XRD patterns of centrate, digestate, pellets 2014 and BC 2014.

Aqueous phase

Condensed water, which is released during pellet pyrolysis, originates from all sorts of physically and chemically bounded water and from the reaction water resulting from the thermo-chemical conversion of sludge (Mislej et al 2012). The resulting aqueous phase also contains many water-soluble pyrolysis products and has a high organic load (Mislej et al. 2015, a), which is indicated by a very high chemical oxygen demand value and high content of volatile fatty acids (Table 8). Biodegradation of such water is somewhat inhibited - the ratio COD: BOD5 is less than 2. The reason for that is a very high concentration of ammonia nitrogen (76.1 gL^{-1}), but that could be an advantage from the viewpoint of the ammonium nitrogen recovery possibility, or it could be directly used as a fertilizer.

Table 8. Physico-chemical characterization of aqueous phase.

Parameters	Units	Aqueous phase
Shape	-	Light yellow -colored liquid
COD	$\text{g O}_2/\text{l}$	127.0
BOD5	$\text{g O}_2/\text{l}$	37.5
pH	-	9.5 (T= 19.7)
Electrical Conductivity (25 °C)	mS/cm	121.30
Dry Matter at T = 105 °C	g/L	45.2
VFA	$\frac{\text{g}}{\text{CH}_3\text{COOH}/\text{L}}$	39.7
Total Nitrogen	$\text{N, g}/\text{L}$	76.0
NH4-N	$\text{N, g}/\text{L}$	76.1

CONCLUSIONS

The dry matter content in pelletized sewage sludge is 91 % - 93 %.. It is in the form of 2 mm – 4 mm in size with a bulk density from 650 kgm^{-3} through 700 kgm^{-3} . It contains a high proportion of organic matter and today it is utilised as an additional alternative solid recovered fuel (SRF) in cement kiln. Following the new trends in sewage sludge final treatment, we must focus on the optimum energy-efficient conversion of organic material, utilization of nutrients such as phosphorus, potassium and nitrogen. Optimum recovery of these substances can be achieved by

pyrolysis. Pyrolysis offers a reasonable positive potential of material utilization of sewage sludge. In the pyrolysis process, sewage sludge is thermally transformed into several fractions. Due to the occurrence of the products appearing in different morphological and physicochemical forms, pyrolysis is certainly a promising procedure for processing of dried sewage sludge. This process gives the possibility for further production of several new materials, which can end the status of waste. The quality of produced biochar and pyrolysis oil must be assessed in relation to the conditions under which they can be declared as by-products, not wastes.

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