

Sewage Sludge Ash-Based Biofertilizers as a Circular Approach to Phosphorus: the Issue of Fe and Al in Soil and Wheat and Weed Plants

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Supporting information

Table S1. Elemental composition of SSA-based P-fertilizers.

Element	Unit	<i>AsBm</i>	<i>A_gAf</i>	<i>AB_gAf</i>	<i>AB_g</i>	<i>AB_gBm</i>	<i>AH_g</i>	<i>AH_gBm</i>
P	% mass.	0.176	9.24	7.50	6.10	5.87	8.68	9.55
N		0.255	0.530	3.190	3.82	3.14	2.89	2.87
K		0.487	0.963	0.727	0.846	0.772	1.09	1.16
Ca		0.694	12.513	12.012	11.369	10.725	13.4	14.6
Mg		0.119	2.442	1.176	1.296	1.188	1.54	1.70
S		0.055	1.38	1.50	2.28	1.91	0.56	0.56
Na		0.049	0.338	0.569	0.382	0.366	0.368	0.595
C		0.590	6.71	23.6	17.6	16.1	12.5	13.9
Fe	g kg ⁻¹	1.679	33.0	15.1	15.4	14.4	26.9	29.0
Al		1.774	24.4	13.9	12.8	11.3	23.7	25.5
Zn		0.117	2.00	1.18	1.75	1.56	3.14	3.29
As	mg kg ⁻¹	< 0.5	4.35	2.55	26.5	< 0.5	31.4	20.0
Cd		0.274	0.43	0.24	0.38	0.84	<0.01	0.345
Cr		5.94	135	60.0	114	94.7	54.7	62.9
Cu		55.0	880	398	470	444	778	850
Ni		2.45	57.7	25.1	52.3	44.3	54.8	62.6
Pb		10.4	21.6	11.2	34.0	14.5	19.9	21.8
B		nd	nd	nd	40,5	35,2	71.3	74.1
Ba		nd	nd	nd	245	234	349	382
Co		nd	nd	nd	5,97	5,46	14.0	16.2
Mn		nd	nd	nd	370	346	562	609
Mo		nd	nd	nd	13,3	10,4	35.3	23.7

nd – no data

Table S2. Basic agricultural data for the experiments.

Item	Experiment				
	I	II	III	IV	V
Wheat cultivar	Trappe	Monsoon	Julius	Julius	Monsoon
Previous crop	spring barley	cereal-legume mixture	winter rape	winter rape	winter rape
Soil tillage system	plough tillage	plough tillage	plough tillage	plough tillage	plough tillage
Fertilization					
K ¹ , kg/ha	99.6	83	83	83	83
N ² , kg/ha	100	110	130	120	130
Plant protection	PP–	PP– or PP+	PP+	PP+	PP+
– herbicides	–	–/+	+	+	+
– fungicides	–	–/+	+	+	+
– insecticides	–	–/+	+	+	+
– growth regulators	–	–	+	+	–
Sowing date	25 April 2014	9 April 2015	2 October 2014	15 October 2015	21 April 2016
Harvest date	11 August 2014	11 August 2015	5 August 2015	9 August 2016	12 August 2016

¹potassium chloride, ²ammonium sulphate, +applied, –not applied.

Table S3. Soil characteristics before the start of the experiments.

Experiment	Soil Type	Soil Texture	pH _{KCl}	Total, g kg ^{–1}					
				C	N	P	K	Mg	P
I	Luvisols	sandy clay loam	6.23	8.31	1.30	0.57	2.98	2.07	0.574
II	Luvisols	sandy loam	5.32	8.90	1.35	0.57	2.90	2.01	0.566
III	Luvisols	sandy loam	5.51	8.87	1.36	0.43	3.21	2.25	0.433
IV	Luvisols	sandy loam	5.23	7.15	1.09	0.55	3.30	2.20	0.548
V	Luvisols	sandy clay loam	6.28	8.53	1.42	0.61	2.98	2.02	0.610

Table S4. Precipitation and air temperature during the study period according to the Meteorological Station in Bałczyn, Poland.

Year	Month											
	I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII
Precipitation, mm												
2014				26.1	34.9	72.2	20.4	59.2	30.8	21.3	21.2	56.6
2015	28.5	8.8	46.0	23.4	25.4	43.0	71.0	13.0	51.2	20.8	80.8	80.4
2016	28.7	50.5	20.5	33.1	70.8	66.3	138.6	71.9	17.1	96.3	78.2	77.8
1981–2010	30.1	23.1	30.7	29.8	62.3	72.9	81.2	70.6	56.2	51.2	46.1	42.6
Air temperature, °C												
2014				9.5	13.3	14.8	21.0	17.9	14.5	9.5	4.4	–0.6
2015	0.6	0.3	4.6	7.2	12.1	15.7	18.0	21.3	14.2	6.6	5.1	3.8
2016	–3.8	2.7	3.6	8.8	14.9	18.0	18.5	17.6	14.7	6.9	2.5	1.0
1981–2010	–2.4	–1.6	1.8	7.7	13.2	15.8	18.3	17.7	13.0	8.1	2.8	–1.0

Soil and plant sampling and preparation

Soil

Soil from the 0–30 cm layer was sampled before the start of each experiment and after wheat harvest, using a hand-held twisting probe (Egner's soil sampler). Sampling was performed at evenly spaced points in each plot and a total of about 1 kg of soil was taken from an individual plot. The

collected soil material was dried at room temperature for several days, thoroughly mixed and sieved. Portions of approximately 300 g were then delivered to the accredited chemical laboratory (Accreditation Certificate No. AB 696 issued by the Polish Centre for Accreditation in Warsaw) for elemental analysis.

Wheat grain

Grain samples weighing approximately 1 kg were taken from the volume resulting after combine harvesting from each plot. Out of these samples, portions of about 200 g of grain were weighed and, after cleaning from impurities and weed seeds, sent for chemical analyses.

Wheat straw

Just prior to harvest, wheat plants were picked up manually from two 1-meter-long rows of each plot. The wheat roots were then cut off at a height matching the combine harvester mowing height and the spikes were removed. Straw (i.e. wheat stems with leaves) was cut into pieces of approx. 10 cm in length. The prepared samples were dried at room temperature for several days, and a portion of 50 g was then weighed from each sample and sent for chemical analyses.

Weeds

Weed samples were collected from a randomly designated 0.25 m² (0.5 m x 0.5 m frame) area from each plot prior to wheat harvest. Weed roots were cut off and the above-ground biomass of weeds was dried at room temperature for several days. Afterwards, the samples in their entirety (different mass) were passed for chemical analyses.

Post-harvest residues

Post-harvest residues, i.e. wheat roots and bottom stem segments (stubble), and the residues of weeds that were not removed from the field during combine harvesting) were sampled from each plot after wheat harvest. Soil columns of 0.40 m² and 0.30 m depth were dug up and then washed on sieves to remove soil particles. The plant material samples prepared in this way were dried at room temperature for several days and sent in their entirety for chemical analysis.

Chemical analyses

Soil total carbon (C) and total nitrogen (N) contents were determined using a Vario Macro Cube Elementar (C,H,N) analyzer (Elementar Analysensysteme, Langenselbold, Germany) and D-phenylalanine (C = 65.44%; N = 8.48%) was used as a standard solution.

The contents of other elements in the soil and plant material were determined using an inductively coupled plasma-optical emission spectrometer (ICP-OES). An appropriate mass (0.5 g) of sample materials (plant or soil) was digested in Teflon vessels (microwave oven Milestone MLS-1200) with 5 mL of concentrated 65 mg kg⁻¹ HNO₃ Suprapur™ (Merck) in the case of plant samples, and with 10 mL of aqua regia in the case of soil samples. After mineralization, all samples were diluted to 50 mL. The ICP-OES with a pneumatic nebulizer with an axial view (iCAP Duo Thermo Scientific, USA) was used to determine the content of elements in all digested and diluted samples. Determination of PTE content was carried out with all the principles of measurement traceability and certified reference materials were used to check the quality and metrological traceability. The levels of detection (LOD) for P, K, Mg, Fe and Al for the soil material were: 3.59, 2.55, 1.17, 0.72 and 2.34 mg kg⁻¹, respectively, and for the plant material, the detection levels for Fe and Al were: 0.040 and 0.015 mg kg⁻¹, respectively.