REPORT ON MINERAL NUTRIENT COMPOSITION OF ANALYSED RECYCLING-DERIVED FERTILISERS

(WPT1_Activity 3_Deliverable 3.1)

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List of abbreviations

AN: Ammonium nitrate

AS: Ammonium sulphate

AW: Ammonia water

DM: Dry matter

EC: Electrical conductivity

FW: Fresh weight

IC: Ion chromatography

ICP-OES: Inductive Coupled Plasma-Optical Emission Spectroscopy

LF: Liquid fraction

NWE: North West Europe

OM: Organic matter

RDFs: Recycling-derived fertilisers

RO: Reverse osmosis

SF: Solid fraction

1. Introduction

Europe demands critical attention towards the inappropriate management of nutrients arising from different biomass streams. One of the key steps to ensure environmental security is to close the nutrient loop and thus warrant the efficient use of these nutrients. The transformation of Europe towards a more circular economy can be achieved by recovery and reuse of nutrients from biomass streams like animal manure, sewage sludge and food waste, which can contribute greatly towards improvement of the efficiency of nutrient management.

ReNu2Farm is an Interreg North-West Europe (NWE) project that focusses on nutrient recycling and upscaling from pilot level to farms and fields. Its priority objective is to optimise the reuse of materials and natural resources in NWE. One of the tasks of the project was to characterise several recycling-derived fertilisers (RDFs) from the processing of the above mentioned biomass streams and determine their mineral nutrient composition. 24 RDFs from 20 producers were characterised for total and plant-available nutrient composition by performing specific chemical extractions. The analysed RDFs include ashes, struvite, composts, digestate and its various derivatives, ammonium sulphate, ammonium nitrate, ammonia water, liquid fraction (LF) of manure, pig urine, flowering pots made of manure, and foliar spray rich in keratin made from chicken feathers (Table 1).

The report consists of 5 chapters. After introduction in Chapter 1, Chapter 2 gives a detailed description on the used analytical methods for assessment of RDFs by UGhent, Arvalis and ULimerick, as the methods might differ. Chapter 3 reports on nutrient composition of the tested RDFs in regard to electrical conductivity (EC), pH, macro- and micro-nutrients, plant-available nutrients, and heavy metals. Chapter 4 proposes potential (theoretical) tailor-made blends on the basis of results from Chapter 3 and deliverable WPT2_D1.2 (*Exploring the demand for recycling-derived nutrients and organic matter in regions of Northwest Europe)* that reports on nutrient demands of crops in the NWE region. Finally, Chapter 5 concludes the report.

Table 1. An overview of the collected recycling-derived fertilisers with respect to country, applied technology, source of origin and product type.

No	Country	Product origin	Technology	Product
1	DE	Sewage sludge	Incineration	Ash 1
2	NL	Poultry litter	Incineration	Ash 2
3	LU	Sewage sludge (50%) and green cuttings (50%)	Gasification	Ash 3
4	LU	Sewage sludge (50%) and green cuttings (50%)	Gasification	Ash 4
5	LU	Sewage sludge	Gasification	Ash 5
6	NL	Sewage sludge	P-precipitation (NuReSys)	Struvite 1
7	BE	Food waste	P-precipitation (NuReSys)	Struvite 2
8	NL	Digested household waste	AD + Composting	Compost 1
9	NL	Manure (> 70%) and waste from feed and dairy industry	AD + Mechanical separation + Acidification	P-poor SF of digestate
10	BE	Manure	AD	Raw digestate
11	NL+BE	SF of pig slurry (30%) and poultry manure (70%)	Mechanical separation + Composting	Compost 2
12	BE	SF of pig slurry (70%) and poultry manure (30%)	Mechanical separation + Composting	Compost 3
13	NL+BE	SF of pig slurry (100%)	AD + Mechanical separation + Composting	Compost 4
14	BE	Manure	H_2SO_4 scrubbing of air from pig stables	Ammonium sulphate
15	BE	Manure (74%), SF of manure (17%) and food waste (9%)	AD + Mechanical separation + Stripping + HNO_3 scrubbing	Ammonium nitrate
16	BE	Manure (45%) and biological waste streams (55%)	AD + Mechanical separation + Evaporation	Ammonia water 1
17	BE	Manure	Source separation of manure via VeDoWS stabling system	Pig urine
18	BE	Digested food waste	AD + Mechanical separation + RO of LF	Mineral concentrate
19	BE	Food and other organic wastes	AD + Mechanical separation + Evaporation + Stripping	Ammonia water 2
20	BE	Food and other organic wastes	AD + Mechanical separation + Evaporation	Concentrate after evaporation
21	BE	Different organic wastes	AD + Mechanical separation	LF of digestate
22	BE	Manure	Mechanical separation	LF of manure
23	BE	Manure	Drying in high temperature ovens	Flowering pots
24	BE	Poultry feathers	Hydrothermal hydrolysis	Keratin-rich foliar spray

AD: anaerobic digestion; RO: Reverse osmosis; SF: solid fraction; LF: liquid fraction

2. Materials and methods

The different analyses performed on the RDFs included pH, electrical conductivity (EC), dry matter (DM), organic matter (OM), total elemental analysis for macro- and micronutrients like nitrogen (N), phosphorus (P), potassium (K), carbon (C), sulphur (S), calcium (Ca), magnesium (Mg), sodium (Na), and for heavy and trace metals like aluminium (Al), cadmium (Cd), chromium (Cr), copper (Cu), cobalt (Co), iron (Fe), manganese (Mn), nickel (Ni), lead (Pb), zinc (Zn), and organic C. Plant-available nutrient analyses were performed for N, P, K, S, Ca, Mg, Na, Fe, Cu, Zn, and chlorine (Cl).

For ash and struvite RDFs (more specifically Ash 1, 2, 5, and Struvite 1 and 2) additional analyses were performed by external certified laboratory 'Innolab' (Oostkamp, Belgium) due to initially observed discrepancies in results provided by involved ReNu2Farm partners. In general discrepancies between results of different laboratories are possible due to lack of standard procedures for the wide array of RDFs, which often can lead to the need for re-analyses. The methods used for each analysis are described briefly in this chapter.

2.1. pH

Method used at UGhent

pH refers to the alkalinity or acidity of a product. pH of the solid RDFs (DM > 13 %) was measured in both, KCl and in water. For pH-KCl, a suspension of samples was prepared in 1M potassium chloride (KCl) solution. 10g of the solid RDF was weighed and added to a beaker, and subsequently mixed with 25mL of 1M KCl. This suspension was stirred and allowed to equilibrate for 10 minutes, after which pH was measured using a pH meter (Orion Star A211, Thermo Fisher Scientific, USA). In the case of pH-water, 10g of the solid RDF sample was mixed with 50 mL demineralised water and allowed to equilibrate for 16 hours, after which the pH of the suspension was measured using the same pH electrode. pH of liquid RDFs (DM < 13%) was measured directly.

Method used at Arvalis / AUREA

For Arvalis, the characterisation of RDFs was performed by AUREA Agrosciences. Water pH was measured by the laboratory as per the French norms[1] number NF EN 13037 or NFU 44172.

2.2. Electrical conductivity (EC)

EC is the ability of a material to transmit or conduct electrical current. It gives an indication of the amount of salts present in the product. The EC was measured on an EC meter (WTW Tetra Con 96, Xylem Analytics, Germany) by preparing water extract of the solid RDFs. 10g of the air-dried RDF was mixed with 50 mL of distilled water and placed on a shaker for 60 minutes. Afterwards, the solutions were filtered using a Whatman filter paper of pore size 125 mm and the extracts were used for the EC determination. The EC of liquid RDFs was measured directly.

2.3. Dry matter (DM)

Method used at UGhent

Samples were weighed in aluminium trays and dried in an oven at 105°C until the stabilisation of weight, after which they were weighed again to determine the DM. For Ash 1, 2, 5, and Struvite 1 and 2, Innolab used the method on the basis of norm BAM/deel 3/03 - Gravimetry[2].

Method used at Arvalis / AUREA

Dry matter was measured according to the European and French norm: NF EN 13040.

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[1] The Norme Francaise ((NF) French Norm) is approved by the Association Française de Normalisation (AFNOR) and the European Norm (EN) is approved by Comité Européen de *Normalisation ((CEN) European Committee for Standardization).*

[2] BAM : Compendium voor bemonsterings - Bodem en veevoeder (Compendium for sampling manure, soil and animal feed)

Method used at ULimerick/ Teagasc Johnstown

A sample aliquot of 15 - 20 g was weighed in porcelain cups and dried for about 20 h at 105°C in an oven, after which they were weighed again to determine the DM.

2.4. Organic matter (OM)

Method used at UGhent

Approximately, 2g of the oven-dried (at 105°C) sample was weighed in a porcelain cup and put in a muffle furnace (Nabertherm, Germany) for 4 hours at 550°C. Afterwards, the samples were weighed to determine their OM content. For Ash 1, 2, 5, and Struvite 1 and 2, Innolab used their own method based on CMA/2/II/A.2 - Gravimetry[3].

Method used at Arvalis / AUREA

The OM was determined according to the European and French norm: NF EN 13039.

Method used at ULimerick/ Teagasc Johnstown

The OM was determined by ignition of the dried residue at 550°C in a muffle furnace for 1 hour followed by weighing of the leftover ashes.

2.5. Total elemental analysis

Performing elemental analysis helps in determining the total elemental composition of the different nutrients present in the RDFs. Except N and C, all other elements were analysed using the Inductive Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), (Varian Vista MPX CCD-Simultaneous, USA).

[3] CMA : Compendium voor monsterneming en analyses van afvalstoffen en bodem (Compendium for sampling and analysis of waste and soil)

2.5.1. Analysis of N

Methods used at UGhent

Analysis of the total N in ashes, struvite, P-poor solid fraction, composts, flowering pots made of manure, and foliar spray made of chicken feathers, was done by using CN analyser (VARIO-MAX, Elementar, Germany and Primacs100, Skalar, the Netherlands). For ash produced from pure sewage sludge via gasification, ammonium sulphate, ammonium nitrate, pig urine, mineral concentrate, ammonia water, concentrate after evaporation, liquid fraction of manure, and liquid fraction of digestate, the total N was analysed by using the Kjeldahl digestion followed by distillation using a Kjeltec-1002 distilling unit (Gerhardt Vapodest, Germany and Kjeltec, FOSS, Denmark). Two different methods were used because it was observed for some products that better destruction/digestion process was achieved with Kjeldahl digestion rather than with the CN analyser. For Ash 1, 2, 5, and Struvite 1 and 2, Innolab used the method based on the norm BAM/deel 3/04 - Titrimetry.

Method used at Arvalis / AUREA

Dumas N was measured according to the European and French norm: NF EN 13654-2.

Method used at ULimerick/ Teagasc Johnstown

Total N in RDFs was analysed by high temperature combustion method using LECO TruSpec CN analyser.

2.5.2. Analysis of C

Method used at UGhent

Total C was estimated using the CN analyser (VARIO-MAX, Elementar, Germany). To measure organic C, total and inorganic C were first analysed using a CN analyser (Primacs100, Skalar, the Netherlands), and organic C was then calculated from these values. For Total C of Ash 1, 2, 5, and Struvite 1 and 2, Innolab used the Dumas method and for inorganic C: Macherey Nagel ref-985064-66 - Spectrophotometry.

Method used at Arvalis / AUREA

Here, organic C was determined and not total C. It was measured according to the European and French norm: NF EN 13039.

Method used at ULimerick / Teagasc Johnstown

Total C in RDFs was analysed by high temperature combustion method using LECO TruSpec CN analyser.

2.5.3. Analyses of P, K, S, Ca, Mg, Na, heavy and trace metals

Method used at UGhent

All the other macro- and micronutrients, and heavy and trace metals, were analysed by preparing extractions of the RDFs in concentrated nitric acid (HNO_3). 0.2 - 0.3g of solid (dried at 105°C) and 4 - 5g of liquid samples were weighed into the microwave tubes. 10mL of 65% HNO_3 was added into the tubes and placed aside for 30 minutes. The tubes were then placed in a sonicator for 15-20 minutes and then in the closed microwave at 180°C. The microwave runs for a total of 60 minutes, where 20 minutes each are spent in three stages : first in ramping the temperature to 180°C, then for holding at 180°C, and finally for cooling down of the tubes. The content of the tubes was filtered using a Whatman filter paper of pore size 125mm into a 50mL volumetric flask pre-rinsed with 1% HNO_3. These extracts were analysed on the ICP-OES (Varian Vista MPX, USA) to determine the total elemental content. For Ash 1 ,2, 5, and Struvite 1 and 2, Innolab used the method based on the norm EPA 3051A - ICP -AES[4].

[4] EPA : Environmental Protection Agency. United States of America.

Method used at Arvalis /AUREA

The oxide forms of macro-nutrients, i.e. phosphorus pentoxide (P_2O_5), potassium oxide (K_2O), calcium oxide (CaO) and magnesium oxide (MgO) were measured according to a method developed by AUREA, adapted from NF EN 13650, dosage from FN EN ISO 11885.

Method used at ULimerick/ Teagasc Johnstown

The concentrations of nutrient elements (P, K, Mg, S, Na, Ca), and metals (As, Cd, Cr, Cu, Ni, Pb, Zn, Al, Fe, Co, Mo and Mn) were determined by an Agilent 5100 synchronous vertical dual-view inductively coupled plasma optical emission spectrometer (Agilent 5100 ICP-OES) following the microwave-assisted acid digestion of required RDF samples. 0.5g of solid (freeze-dried, ball-milled in case of slurry or sludge) samples were weighed in the microwave tubes. 2ml deionised water followed by 2.5ml HCl and 7.5ml of HNO_3 was added into the tubes and left open for 15 minutes to pre-digest in a fume-hood. The tubes were then capped tightly and positioned on the microwave digestion carousel to transfer into the microwave and run for a total of 60 minutes, where 20 minutes each were spent in ramping the temperature to 180°C, holding at 180°C and cooling down of the tubes. When digestion is complete and cool down is over, the tubes were left in a fume-hood to vent for 15-20 minutes. Then, the contents of the tubes were filtered using a Whatman filter paper No. 42 or equivalent filter paper into a clean 50mL volumetric flask. Digested, filtered samples were stored in screw-top plastic containers prior to analysis.

2.6. Plant-available nutrient analyses

Plant-available nutrients analysed for this report include those found in inorganic forms that are available for the plant uptake.

2.6.1. Analysis of plant-available N

Method used at UGhent

The plant-available forms of N are ammonium-N (NH_4-N) and nitrate-N (NO_3-N). The RDFs were extracted in different ratios in 1M KCl solution to analyse the NH_4-N and NO_3-N. The extracts were then analysed using a flow auto-analyser (Chemlab System 4, Skalar, the Netherlands). For Ash 1, 2, 5, and Struvite 1 and 2, Innolab used the method based on the norm BAM/deel 03/05 - Titrimetry for NH_4-N and Machery-Nagel re 985064-66 - Spectrophotometry for NO_3-N.

Method used at Arvalis / AUREA

NH_4-N and NO_3-N were measured by a method developed by AUREA, using KCL extraction.

2.6.2. Analysis of plant-available P

The analysis of plant-available P was done by two different extractions as reported below: water extraction and extraction using neutral ammonium citrate.

2.6.2.1. Water extraction

0.4 - 0.5g of solid (dried at 105°C) and 4 - 5g of liquid samples were mixed with 50mL distilled water and placed on a shaker for 30 minutes. The samples were filtered using a Whatman filter paper of pore size 125mm, after which 1 drop of 65% HNO_3 was added to the filtrate for sample stability. These samples were analysed on the ICP-OES. These same extracts were also used to analyse plant-available K, S, Ca, Mg, Na, Fe, Cu, and Zn

2.6.2.2. Extraction using neutral ammonium citrate

A prepared solution of ammonium citrate at pH 7 was added to 0.8 - 1g of solid samples (dried at 105°C) and placed in a water bath at 65°C for 60 minutes with occasional shaking of the flasks. After 60 minutes, the flasks were removed out of the water bath and placed aside to cool down and their content was filtered using a Whatman filter paper of pore size 125mm. 1 drop of 65% HNO_3 was added to the filtrate for sample stability. These samples were analysed on the ICP-OES. For Ash 1, 2, 5, and Struvite 1 and 2, Innolab used the method based on the Joulie and Petermann's ammonium citrate extraction.

2.6.3. Analysis of plant-available Cl

Plant-available form of Cl is the chloride ion (Cl^-). The method of analysis of Cl^- is different for solid and liquid samples. Liquid samples are analysed using ion chromatography (IC) (761 Compact IC, Metrohm, Switzerland). For measurement of Cl^-, the EC of the RDFs is an important parameter to be considered. If the EC < 300 µS/cm, the samples are filtered through a 0.45 µm membrane filter and the filtrate is used for analysis. The samples can also be centrifuged if the particle content in them is very high. For RDFs with an EC > 300 µS/cm, the filtrate is diluted using milliQ water after filtration using 0.45 µm membrane. For solid RDFs, potentiometric titration was used to estimate the Cl^- in the products. The analysis of Cl^- is carried out in a suspension of sample in 0.15M HNO_3 by titration with silver nitrate ($AgNO_3$) and potentiometric determination of the equivalence point using couple reference ($Hg/HgSO_4$) and indicator ($Ag/AgCl$) electrodes.

3. Nutrient composition of RDFs

This chapter compiles results of all the analyses performed on the collected RDFs.

3.1. Physicochemical and elemental analysis

Results of pH, EC, DM and OM for the RDFs are reported in Table 2. It was observed that the pH-KCl of ash 5 had a value higher than 14. Lowest pH-KCl in case of solid RDFs was observed for P-poor SF (pH-KCl = 5.2), which is probably result of applied acidification in order to remove P. These same RDFs showed highest and lowest pH respectively, when measured with water as the extractant. Among liquid RDFs, both ammonia waters showed the highest pH value of 11 and the mineral concentrate had the lowest pH of 2.3. The low pH is a result of the addition of sulphuric acid during the reverse osmosis step from which the mineral concentrate was derived. Ammonia water 2 had the highest EC of 312 mS/cm among all the RDFs, whereas, the lowest EC of 0.2 mS/cm was measured in ash 3. Ash 1, 3, and 5 contained no moisture, whereas LF of manure had the lowest DM of 1.6%. The keratin-based foliar spray contained 94% OM, i.e. highest among the tested RDFs, and ash 1 had the lowest OM of 0.01%. Differences in OM results were observed between UGhent and Arvalis / AUREA's analysis for struvite 1, and compost 3. These differences could be owed to the difference in batches of products that were analysed and/or the analysis done at different period interval during the product storage (as done by Arvalis / AUREA). It could also be a result of different methodology used.

Total C, OC and total N results are presented in Table 3. Highest total C and OC were observed in flowering pots made of manure with a range of 385-484 g/kg for total C and a mean value of 410 g/kg for OC. In the case of total N, ammonia water 2 had the highest N content of 155 g/kg.

Results for total elemental analyses of P, K, S, Ca, Mg and Na are given in Table 4. In the analyses by UGhent, it was seen that total P was highest in struvite 1 (128 g/kg) which was also mirrored by the analysis of Arvalis / AUREA, where struvite 1 had a similar total P (124 g/kg).

Among all the RDFs, highest total K was seen in ash 2 (94 g/kg). In case of total S, highest values were observed in ash 1 at 50 g/kg. Ash 5 exhibited the highest Ca (135 g/kg by UGhent and 215 by Arvalis / AUREA), whereas highest Mg was observed in the struvites (struvite 1: 105 g/kg and 90 g/kg in analysis by UGhent and ULimerick / Teagasc Johnstown respectively; struvite 2: 103 g/kg and 99 g/kg in analysis by UGhent and ULimerick / Teagasc Johnstown respectively). Ash 1 displayed the highest Na content (118 g/kg in analysis by UGhent and 100 g/kg in analysis by ULimerick / Teagasc Johnstown).

Table 2. Mean and standard deviation of pH, EC, dry matter (DM) and organic matter (OM) (based on DM) in tested products. Results of analyses performed by Arvalis are reported in ranges as they were carried out few times on different occasions in time (e.g. before and during fertilisation in ReNu2Farm field trials). Results of flowering pots are also mentioned in ranges since the tested pots were of different type (i.e. based on different percent of input material, moisture content, etc.) as provided by the producer.

Product	**Project partner**	**pH-KCl (solids)**	**pH-water (solids)**	**pH (liquids)**	**EC (mS/cm)**	**DM (%)**	**OM (% DM)**
Ash 1	UGhent	11	10	-	32	100±0	0.01
Ash 1	ULimerick	n.d.	n.d	-	n.d.	100	n.d.
Ash 2	UGhent	12	12	-	41	94	3.8
Ash 2	Arvalis	n.d	13	-	n.d.	94	2.8
Ash 2	ULimerick	n.d.	n.d.	-	n.d.	100	n.d.
Ash 3	UGhent	10	9.5	-	0.2	100±0	0.31±0.2
Ash 4	UGhent	8.8	8.8	-	41	89±0.86	12±0.17
Ash 5	UGhent	>14	13	-	9.9	100±1.4	5.5
Ash 5	Arvalis	n.d.	13	-	n.d.	99	7.8
Struvite 1	UGhent	7.1	7.9	-	0.82	61	27
Struvite 1	Arvalis	n.d.	8.7	-	n.d.	55	0.84
Struvite 1	ULimerick	n.d.	n.d.	-	n.d.	51	18
Struvite 2	UGhent	8.8	8.7	-	0.85	56	16
Struvite 2	ULimerick	n.d.	n.d.	-	n.d.	58	20
Compost 1	UGhent	9.1	8.9	-	12	65±0.27	70±0.83
P - poor solid fraction of digestate	UGhent	5.2	6.2	-	1.1	39±1.5	90±0.13
Raw digestate	UGhent	8.9	9.1	-	5.1	16±0.38	73±0.19
Compost 2	UGhent	8.5	9.1	-	4.8	33±0.33	75±0.59
Compost 2	Arvalis	n.d.	8.4-8.8	-	n.d.	33-37	73
Compost 3	UGhent	8.9	8.9	-	13	56±0.25	62±0.9
Compost 3	Arvalis	n.d.	8.6-8.8	-	n.d.	33-63	26-42
Compost 4	UGhent	8.7	8.4	-	10	50±0.4	62±0.97
Compost 4	Arvalis	n.d.	8.6-9.1	-	n.d.	32- 63	66-68
Ammonium sulphate	UGhent	-	-	6.1	165	25±1.6	n.d.

Ammonium nitrate	UGhent	-	-	5.7	303	23±0.05	n.d.
Ammonia water 1	UGhent	-	-	11	187	n.d.	n.d.
Pig urine	UGhent	-	-	8.7	48	2.2±0.02	36±0.92
Mineral concentrate	UGhent	-	-	2.3	34	2.8±0.38	55±1.1
Ammonia water 2	UGhent	-	-	11	312	n.d.	n.d.
Concentrate after evaporation	UGhent	9.8	9.1	-	15	13±0.11	62±0.90
Liquid fraction of digestate	UGhent	-	-	8.4	37	2.0±0.01	35±0.81
Liquid fraction of manure	UGhent	-	-	8.0	27	1.6±0.15	41±0.75
Flowering pots made of manure	UGhent	6.8	7.2	-	2.8-3.9	96±0.66	87±2.6
Keratin-based foliar spray	UGhent	8.0	n.d.	-	n.d.	34±1.2	94±0.21

n.d. = not determined

Table 3. Total and organic carbon (C), and total nitrogen (N) on fresh weight basis. Results of analyses performed by Arvalis are reported in ranges as they were carried out few times on different occasions in time (e.g. before and during fertilisation in ReNu2Farm field trials). Results of flowering pots are also mentioned in ranges since the tested pots were of different type (i.e. based on different percent of input material, moisture content, etc.) as provided by the producer.

Product	**Project partner**	**Total C (g/kg)**	**Organic C (g/kg)**	**Total N (g/kg)**
Ash 1	UGhent	6.5	<0.2	<0.16
Ash 1	ULimerick	1.3	n.d.	0.25±0.18
Ash 2	UGhent	15	6.4	<0.16
Ash 2	Arvalis	n.d.	1.7	0.4
Ash 2	ULimerick	8.9	n.d.	0.15±0.05
Ash 3	UGhent	<5.5	0.2±0	<0.9
Ash 4	UGhent	64	52±0.03	<0.9
Ash 5	UGhent	101	10	3.0
Ash 5	Arvalis	n.d.	39	2.4

Struvite 1	UGhent	16	0.53	55
Struvite 1	Arvalis	n.d.	2.3	57
Struvite 1	ULimerick	1.3	n.d.	51±0.21
Struvite 2	UGhent	13	0.46	51
Struvite 2	ULimerick	1.6	n.d.	51±0.25
Compost 1	UGhent	231	190±0.53	26
P - poor solid fraction of digestate	UGhent	167	130±0.35	5.8
Raw digestate	UGhent	140	36±0.4	5.2
Compost 2	UGhent	194	120±0.36	14.4
Compost 2	Arvalis	n.d.	120-130	16-18
Compost 3	UGhent	220	210±1.9	22.3
Compost 3	Arvalis	n.d.	110-210	13-28
Compost 4	UGhent	170	160±2	17
Compost 4	Arvalis	n.d.	110-140	9.5-17
Ammonium sulphate	UGhent	<5.5	n.a.	39
Ammonium nitrate	UGhent	<5.5	n.a.	82
Ammonia water 1	UGhent	<5.5	n.a.	107
Pig urine	UGhent	6.8	n.a.	6.1
Mineral concentrate	UGhent	<5.5	n.a.	3.2
Ammonia water 2	UGhent	<5.5	n.a.	155
Concentrate after evaporation	UGhent	33	31±0.05	4.6
Liquid fraction of digestate	UGhent	5.7	0.2±0	4.6
Liquid fraction of manure	UGhent	<5.5	0.3±0.02	3.3
Flowering pots made of manure	UGhent	385-454	410±0.53	4.0-8.9
Keratin-based foliar spray	UGhent	185±36	n.d.	24±10

n.d. = not determined; n.a. = not applicable, i.e. values for IC not detected, hence not possible to calculate OC

Table 4. Mean and standard deviation of total P, K, S, Ca, Mg, and Na on fresh weight basis. Results of analyses performed by Arvalis are reported in ranges as they were carried out few times on different occasions in time (e.g. before and and during fertilisation in ReNu2Farm field trials). Results of flowering pots are also mentioned in ranges since the tested pots were of different type (i.e. based on different percent of input material, moisture content, etc.) as provided by the producer.

Product	**Project partner**	**Total P (g/kg)**	**Total K (g/kg)**	**Total S (g/kg)**	**Total Ca (g/kg)**	**Total Mg (g/kg)**	**Total Na (g/kg)**
Ash 1	UGhent	65	10	50	76	11	118
Ash 1	ULimerick	84	13±0.05	30±1.1	103±0.93	15±0.23	100±1.3
Ash 2	UGhent	53	95	26	166	29	13
Ash 2	Arvalis	55	134	n.d.	153	33	n.d.
Ash 2	ULimerick	55	107±3.9	31±1.2	156±27	35±1.9	14±0.79
Ash 3	UGhent	5.1±1.4	8.6±0.11	6.8±0.21	4.0±0.16	0.7±0.16	0.72±0.3
Ash 4	UGhent	3.0±0.06	16±1.0	11±0.33	67±2.0	4.3±0.35	39±4.1
Ash 5	UGhent	67	4.7	24	135	9.9	3.0
Ash 5	Arvalis	64	2.7	n.d.	215	9.6	n.d.
Struvite 1	UGhent	148	0.60	0.04±0	0.093	105	0.22
Struvite 1	Arvalis	124	<0.83	n.d.	<3.5	90	n.d.
Struvite 1	ULimerick	26	0.58±0.03	0.03±0.01	0.27±0.01	94±2.8	0.01±0
Struvite 2	UGhent	153	19	0.14±0.06	0.27	103	0.31
Struvite 2	ULimerick	30	12±0.07	0.06±0.01	0.43±0.14	99±0.88	0.1±0.01
Compost 1	UGhent	8.2±1.1	15±3.0	6.7±0.79	23±3.1	2±0.34	2.2±0.27

P - poor solid fraction of digestate	UGhent	1.5±0.56	0.28±0	5.33±1.56	6.5±0.34	0.47±0.07	0.13±0.01
Raw digestate	UGhent	2.5±0.21	3.7±0.08	1.29±0.07	4.4±0.01	1.5±0.06	1.8±0
Compost 2	UGhent	6.9±0.02	7.0±0.45	4.2±0.14	9.0±0.18	5.8±0.02	1±0.03
Compost 2	Arvalis	8±0	9.5-12	2.5	12-16	5.2-5.8	1.4
Compost 3	UGhent	6.7±1.1	7.5±2.7	4.9±1.0	<0.013	5.0±0.71	1.9±0.29
Compost 3	Arvalis	6-10	9-12	7.4	12-30	5.1-5.8	2.8
Compost 4	UGhent	6.5±1.8	16±0.25	5.1±0.51	6.8	3.6±0.86	3.1±0.05
Compost 4	Arvalis	7-14	6.5-21	6.7	8-18	5.7-8.5	6.9
Ammonium sulphate	UGhent	0.004±0	0.12±0.01	28.4±1.5	0.14±0.01	<0.00031	0.31±0.01
Ammonium nitrate	UGhent	<0.00038	0.01±0	0.03±0	0.02±0.01	<0.00031	0.02±0
Ammonia water 1	UGhent	<0.00038	<0.0023	0.28±0	<0.00063	<0.00031	<0.0017
Pig urine	UGhent	<0.00038	3.5±0.09	0.75±0.08	0.01	0.06	1.2±0.03
Mineral concentrate	UGhent	0.1±0	2.3±0.04	5.4±0.11	0.1±0	0.1±0	2.1±0.03
Ammonia water 2	UGhent	<0.00038	<0.0023	1.4±0.05	<0.00063	<0.00031	<0.0017
Concentrate after evaporation	UGhent	1.0±0.04	10±0.4	1.3±0.05	0.88±0.02	0.08±0	15±0.54
Liquid fraction of digestate	UGhent	0.26±0	3.3±0.08	0.16±0	0.13±0	0.01±0	0.71±0
Liquid fraction of manure	UGhent	0.06±0	2.7±0.02	0.21±0	0.22±0	0.05±0	0.39±0.02
Flowering pots made of manure	UGhent	1.2-2.2	2.4-4.3	1.2-1.9	1.6-2.4	1.2-2.1	0.87-1.2
Keratin-based foliar spray	UGhent	0.58±0.03	0.78±0.05	3.9±0.05	0.59±0.06	0.09±0.01	4.6±0.95

n.d. = not determined

3.2. Heavy metals

The RDFs were analysed for heavy metals Zn, Fe, Cu, Al, Cr, Mn, Ni, Co, Cd, Pb, As and Mo (Table 5 and 6). In general, higher metal content was observed in the ashes in comparison to other RDFs. Table 7 provides legal limits set on these elements by the EU regulations (Regulation (EU) 2019/1009). For the purpose of comparison with legal limits, the results for heavy metals are expressed on dry matter basis.

Highest Zn content was detected in ash 5 (1991 mg/kg), which is higher than the legal limit of 1500 mg/kg for inorganic fertilisers. Ash 1 had the highest Cu (426 mg/kg in analysis by UGhent and 609 mg/kg in analysis by ULimerick / Teagasc Johnstown) content. ULimerick / Teagasc Johnstown's analysis gave a value higher than the legal limit of 600 mg/kg. Ash 4 had very high Ni (4812 mg/kg) and Pb (232 mg/kg) content in comparison to the legal limit of 100 mg/kg and 120 mg/kg respectively, for inorganic fertilisers. Since ash 5 was produced from incineration of solely sewage sludge as feedstock, it is observed to have a higher metal content for some analysed metals. Hence, the producer currently provides this ash to the construction sector, even though it contains high amounts of plant nutrients like P and K. In the case of metals like Zn, Cu, Fe and Al, the general trend observed is that, the ashes obtained from incineration (ash 1 and 2) show higher total values, in comparison to the ashes obtained from gasification process. The LF of manure had the highest Cd content of 10 mg/kg which is higher than the legal limit of 1.5 mg/kg for organic fertilisers. The LF of manure has the lowest DM content (< 2%), and by expressing heavy metals on DM basis an exceedance is expected. In practice, however, contribution of heavy metals via application of LF of manure is expected not to have detrimental effect on the environment as the absolute amount of added heavy metals should be low. Arvalis / AUREA tested compost 4 for As and ULimerick / Teagasc Johnstown tested ashes 1 and 2, and both the struvites for As and Mo. Both metals were found to be lower than the legal limits.

Table 5. Mean and standard deviation of total Zn, Fe, Cu, Al, and Cr in the tested products on dry matter basis. Results of flowering pots are mentioned in ranges since the pots tested were of different types (based on percent of input material/moisture content etc.) as provided by the producer. Results of flowering pots are also mentioned in ranges since the tested pots were of different type (i.e. based on different percent of input material, moisture content, etc.) as provided by the producer.

Product	Project partner	Total Zn (mg/kg)	Total Fe (mg/kg)	Total Cu (mg/kg)	Total Al (mg/kg)	Total Cr (mg/kg)
Ash 1	UGhent	1228	43626	426	45423	73
Ash 1	ULimerick	1797±34	59622±766	609±4.0	52980±296	112±3.4
Ash 2	UGhent	1417	3490	296	4850	14
Ash 2	ULimerick	1940±43	4633±175	417±3.7	7459±1228	20±1.5
Ash 3	UGhent	48±9.9	5350±1480	9.3±2	2604±670	155±19
Ash 4	UGhent	1061±123	44952±6095	278±1	14090±499	7037±966
Ash 5	UGhent	1991	143366	401	18319	76
Struvite 1	UGhent	<4.6	437	0.73	11	1.9
Struvite 1	ULimerick	4.4±5.3	277±10	0.32±0.14	35±3.3	2.3±0.1
Struvite 2	UGhent	<4.0	25	0.70	36	1.7
Struvite 2	ULimerick	4.1±0.54	62±9.1	0.49±0.06	40±3.1	2.8±0.06
Compost 1	UGhent	374±21	3692±79	90±2.8	1246±11	14±1.7
P - poor solid fraction of digestate	UGhent	200±6.2	2506±213	72±3.8	395±11	15±1.4
Raw digestate	UGhent	485±5.9	5714±32	228±4.4	831±42	14±2.4
Compost 2	UGhent	377±2.8	1148±73	102±1	516±20	5.3±0.3
Compost 3	UGhent	480±1.9	4096±1023	106±1.8	1540±139	15±0.17
Compost 4	UGhent	502±32	12873±211	119±88.2	1764±0	22±3.1
Compost 4	Arvalis	92	n.d.	70	n.d.	24
Ammonium Sulphate	UGhent	4.5±0.18	3.5±0	0.12±0	1.9±0.01	0.23±0.04

Ammonium nitrate	UGhent	0.71±0.08	1.6±0.05	0.01±0	1.9±0.83	0.25±0.02
Ammonia water 1	UGhent	0.98±0.09	0.72±0	0.23±0.13	0.83±0.14	<0.13
Pig urine	UGhent	0.55±0.07	2.0±0.17	0.25±n.a.	0.37±0.52	0.14±0.02
Mineral concentrate	UGhent	0.76±0.03	157±0	0.10±0.01	<0.5	1.1±0.02
Ammonia water 2	UGhent	<0.31	<0.31	<0.25	<0.5	<0.13
Concentrate after evaporation	UGhent	25±1.6	768±0.93	<5	42±1	145±14
Liquid fraction of digestate	UGhent	3.1±0.3	3.05±0.49	0.74±0.06	8.0±0.08	0.28±0.04
Liquid fraction of manure	UGhent	22±0.21	22±1.8	13±0.01	10±0.07	0.31±0
Flowering pots made of manure	UGhent	39-63	572-1728	5.3-18	357-517	60-187
Keratin-based foliar spray	UGhent	5.3±1	15±2.2	<5	4.7±0.36	<2.5

n.d. = not determined

Table 6. Mean and standard deviation of total Mn, Ni, Co, Cd, Pb, As, and Mo in the products on dry matter basis. Results of flowering pots are mentioned in ranges since the tested pots were of different type (i.e. based on different percent of input material, moisture content, etc.) as provided by the producer.

Product	**Project partner**	**Total Mn (mg/kg)**	**Total Ni (mg/kg)**	**Total Co (mg/kg)**	**Total Cd (mg/kg)**	**Total Pb (mg/kg)**	**Total As (mg/kg)**	**Total Mo (mg/kg)**
Ash 1	UGhent	692	45	7.6	3.4	11	n.d.	n.d.
Ash 1	ULimerick	955±4.8	59±1.9	12±0.56	0.25	20±0.99	<1.5	15±0.63
Ash 2	UGhent	1417	17	2.5	0.69	7.3	n.d.	n.d.
Ash 2	ULimerick	1915±45	22±0.52	2.5±0.51	0.98±0.09	37±44	<1.5	12±2.4
Ash 3	UGhent	147±46	345±61	<13	4.6±0	<63	n.d.	n.d.
Ash 4	UGhent	6008±351	4812±577	40±5.7	5.8±0.97	232±272	n.d.	n.d.

Ash 5	UGhent	437	57	16	6.7	7.2	n.d.	n.d.
Struvite 1	UGhent	31	0.18	<0.093	<0.093	<0.093	n.d.	n.d.
Struvite 1	ULimerick	49±1.6	<0.6	<0.3	<0.15	<2	<1.5	<0.5
Struvite 2	UGhent	93±0.17	0.089	<0.079	<0.079	<0.079	n.d.	n.d.
Struvite 2	ULimerick	128±1.1	<0.6	<0.3	<0.15	<2	<1.5	<0.5
Compost 1	UGhent	359±39	<25	<13	<3.3	<63	n.d.	n.d.
P - poor solid fraction of digestate	UGhent	146±0.4	<25	<13	<3.3	<63	n.d.	n.d.
Raw digestate	UGhent	425±2.8	<25	<13	<3.3	<63	n.d.	n.d.
Compost 2	UGhent	625±1.7	<25	<13	<3.3	<63	n.d.	n.d.
Compost 3	UGhent	433±14	<25	<13	<3.3	<63	n.d.	n.d.
Compost 4	UGhent	572±31	<25	<13	<3.3	<63	n.d.	n.d.
Compost 4	Arvalis	n.d.	7.5	n.d.	<0.12	<3.0	<0.6	n.d.
Ammonium Sulphate	UGhent	3.2±0.04	<1.3	<0.63	<0.16	<3.1	n.d.	n.d.
Ammonium nitrate	UGhent	1.6±0.08	<1.3	<0.63	<0.16	<3.1	n.d.	n.d.
Ammonia water 1	UGhent	0.48±0.05	<1.3	<0.63	<0.16	<3.1	n.d.	n.d.
Pig urine	UGhent	1.8±0.34	<1.3	<0.63	<0.16	<3.1	n.d.	n.d.
Mineral concentrate	UGhent	564±5.3	<1.3	<0.63	<0.16	<3.1	n.d.	n.d.
Ammonia water 2	UGhent	<0.16	<1.3	<0.63	<0.16	<3.1	n.d.	n.d.
Concentrate after evaporation	UGhent	24±1.1	3.1±0	<13	<3.3	<63	n.d.	n.d.
Liquid fraction of digestate	UGhent	1.7±0.08	<1.3	<0.63	<0.16	<3.1	n.d.	n.d.

Liquid fraction of manure	UGhent	22±0.21	22±1.8	13±0.01	10±0.07	<3.1	n.d.	n.d.
Flowering pots made of manure	UGhent	57-99	32	<13	<3.3	<63	n.d.	n.d.
Keratin-based foliar spray	UGhent	<3.3	<25	<13	<3.3	<63	n.d.	n.d.

n.d. = not determined

Table 7. Maximum allowed inorganic contaminants concentration (heavy and trace metals) for different fertiliser category (Regulation (EU) 2019/1009).

Parameter	Organic fertilisers	Organo-mineral fertilisers	Inorganic macronutrient fertilisers	Unit
Cd	1.5	3 (<5% P_2O_5), 60 (>5% P_2O_5)	3 (<5% P_2O_5), 60 (>5% P_2O_5)	mg/kg DM
Ni	50	50	100	mg/kg DM
Pb	120	120	120	mg/kg DM
As	40	40	40	mg/kg DM
Cu*	300	600	600	mg/kg DM
Zn*	800	1500	1500	mg/kg DM

**As declared in the legislation 2019/1009. However, these limit values shall not apply where the copper or zinc has been intentionally added to the fertilising product for the purpose of correcting the soil micronutrient deficiency.*

3.3. Plant-available nutrients

Highest NH_4-N content of 155 g/kg was observed in ammonia water 2. All ashes show very low levels of NH_4-N as expected. Most RDFs had very low levels to below detection limit values of NO_3-N, with ammonium nitrate having the highest content of 43 g/kg NO_3-N. The higher NO_3-N content is due to the addition of nitric acid (HNO_3) as a sorbent in the scrubbing column. Moreover, a column with mineral N/total N (%) is provided enabling to determine the % of total N that can be found in mineral form in each RDF. Ammonium nitrate, ammonium sulphate, ammonia water and struvite 1 have a 100% mineral N/total since all the N present in these products are inorganic in nature. In the case of concentrate after evaporation, mineral N/total N ratio amounts to 96%. Generally, concentrate after evaporation is expected to have higher organic N concentration since the N is evaporated and the condensate is left with nutrients like P and K. In this case, the high content of mineral N can be attributed to the addition of ammonia water to the concentrate at the end of the process chain by the producer, done as a measure to market the RDF as N fertiliser.

For analysis of plant-available P extracted with water, ash 1 and struvite 2, both, exhibited the highest value of 4.4 g/kg, followed closely by struvite 1 with a P content of 3.8 g/kg. However, for the analysis done by extraction in NAC, struvite 2 showed the highest P (153 g/kg) among tested RDFs. Similar to N, a column is provided for mineral P_{NAC} / Total P (%) and it can be seen that ash 1, and both struvites show 100%, i.e. all the P present in these RDFs is in the plant-available form. For products like ammonium sulphate, ammonium nitrate, ammonia water and pig urine, plant-available P analysis by NAC extraction was not performed as their total P levels are below limits of detection.

Other plant-available macro- and micro-nutrients, like K, S, Na, Ca and Mg, and heavy metals like Fe, Cu and Zn were assessed. As with total K, highest plant-available K (79 g/kg) was also observed in ash 2. This implies that all the K present in the ash is in the usable form for plant-uptake which could make it a desirable RDF. Highest S content of 36 g/kg and highest Na content of 56 g/kg was seen in ash 1, corresponding to the highest total S value as reported in Table 4. Ash 5 exhibited highest plant-available Ca content of 31 g/kg, whereas, highest Mg content was seen in struvite 2 (1 g/kg).

In terms of plant-availability of metals, highest Fe (205 mg/kg) was observed in raw digestate, Cu (15 mg/kg) in ash 1 and compost 1, and Zn (51 mg/kg) in compost 3. The ashes where total metal contents were seen to be very high, had lower plant-available values. This indicates that probably the plant uptake of these metals would be low as compared to its presence in the RDF.

Cl content in the RDFs was analysed and ash 4 had the highest content of 42 g/kg of Cl in it. Both struvites had Cl below quantifiable limits.

Table 8. Mean and standard deviation of plant-available N (NH_4 and NO_3), and plant-available P (extracted in water and neutral ammonium citrate (NAC)) on fresh weight basis. Results of analyses performed by Arvalis are reported in ranges as they were carried out few times on different occasions in time (e.g. before and and during fertilisation in ReNu2Farm field trials). Results of flowering pots are also mentioned in ranges since the tested pots were of different type (i.e. based on different percent of input material, moisture content, etc.) as provided by the producer.

Product code	Project partner	NH_4-N (g/kg)	NO_3-N (g/kg)	Mineral N/Total N (%)	P by water extraction (g/kg)	P by NAC extraction (g/kg)	Mineral P_{NAC}/Total P (%)
Ash 1	UGhent	0.157	<0.01	-	4.4	65	100
Ash 2	UGhent	<0.157	<0.01	-	0.73	40	75
Ash 2	Arvalis	<0.1	0.01	-	n.d.	n.d.	n.d.
Ash 3	UGhent	0.01±0.01	<0.002	-	0.02±0	2.3±0.4	45
Ash 4	UGhent	0.02±0	<0.002	-	0.05±0	2.3±0.2	77
Ash 5	UGhent	<0.157	<0.01	-	<0.02	57	84
Ash 5	Arvalis	<0.1	<0.01	-	n.d.	n.d.	n.d.
Struvite 1	UGhent	55	<0.01	99	3.8	148	100
Struvite 1	Arvalis	57	n.d.	100	n.d.	n.d.	n.d.
Struvite 2	UGhent	51	0.01	99	4.4	153	100
Compost 1	UGhent	3.5±0.17	<0.002	18	1.4±0.09	5.5±0.87	67
P - poor SF of digestate	UGhent	0.01±0	0.06±0.09	0.17	0.15±0.02	0.39±0.04	26
Raw digestate	UGhent	2.1±0.25	<0.002	40	0.26±0.01	2.5±0.89	100
Compost 2	UGhent	4.3±0.61	<0.002	33	0.97±0.07	2.3±0.11	33
Compost 2	Arvalis	4.5-6.7	<0.01	28-37	n.d.	n.d.	n.d.
Compost 3	UGhent	2.1±0.01	<0.002	9.4	2.0±0.08	4.8±0.18	72
Compost 3	Arvalis	3.6-5.6	<0.01	20-28	n.d.	n.d.	n.d.
Compost 4	UGhent	0.38±0.01	0.77±0.03	6.8	1.9±0.06	5.0±1.1	77
Compost 4	Arvalis	0.5-1	0.06-0.54	9.1-12	n.d.	n.d.	n.d.

Ammonium Sulphate	UGhent	39±3.0	<0.002	100	0.01±0	n.a.	n.a.
Ammonium nitrate	UGhent	48±2.6	34±0.74	100	<0.0005	n.a.	n.a.
Ammonia water 1	UGhent	107±6.8	<0.002	100	<0.0005	n.a.	n.a.
Pig urine	UGhent	5.1±0.05	<0.002	84	<0.0005	n.a.	n.a.
Mineral concentrate	UGhent	2.7±0.19	<0.002	84	<0.0005	<0.0005	n.a.
Ammonia water 2	UGhent	155±0	<0.002	100	<0.0005	n.a.	n.a.
Concentrate after evaporation	UGhent	4.4±0.3	<0.002	96	0.07±0	0.79±0.26	79
Liquid fraction of digestate	UGhent	3.5±0.05	<0.002	76	<0.0005	<0.0005	n.a.
Liquid fraction of manure	UGhent	2.5±0.06	<0.002	76	0.01±0	<0.0005	n.a.
Flowering pots made of manure	UGhent	0.2-0.3	0.03-0.05	1.3-3.9	n.d.	n.d.	n.d.
Keratin-based foliar spray	UGhent	12±4.1	<0.002	50	n.d.	n.d.	n.d

n.d. = not determined ; n.a. = not applicable

Table 9. Mean and standard deviation of plant-available K, S, Na, Ca, and Mg on fresh weight basis.

Product code	**Project partner**	**K (g/kg)**	**S (g/kg)**	**Na (g/kg)**	**Ca (g/kg)**	**Mg (g/kg)**
Ash 1	UGhent	0.98±0.07	36±0.72	56±0.47	0.47±0.07	0.05±0.01
Ash 2	UGhent	79±0.47	22.94±1.1	8.1±0.2	0.29±0.06	<0.0025
Ash 3	UGhent	<0.046	0.13±0	0.07±0.02	0.19±0.04	<0.0025
Ash 4	UGhent	14±0.04	7.6±0.21	46±3.9	7.2±0.38	0.18±0
Ash 5	UGhent	<0.046	4.2±0.08	0.07±0	31±0.94	<0.0025
Struvite 1	UGhent	<0.046	0.03±0.02	0.03±0	0.02±0	0.77±0.05
Struvite 2	UGhent	0.46±0	0.06±0	0.14±0	0.03±0	1.0±0

Compost 1	UGhent	2.3±0.07	3.4±0.06	1.7±0.04	0.51±0.05	0.35±0.02
P-poor solid fraction of digestate	UGhent	0.05±0.01	0.39±0.04	0.06±0.01	0.28±0.03	0.1±0.02
Raw digestate	UGhent	0.68±0.01	0.03±0.01	0.35±0.01	0.1±0	0.04±0
Compost 2	UGhent	2.8±0.07	0.25±0.01	0.51±0.02	0.17±0.01	0.15±0
Compost 3	UGhent	2.2±0.08	3.7±0.32	1.8±0.17	0.77±0.03	0.5±0.02
Compost 4	UGhent	1.4±0.08	1.4±0	3.7±0.12	0.21±0.01	0.26±0
Ammonium sulphate	UGhent	0.05±0	0.09±0	0.1±0	0.07±0	0.01±0
Ammonium nitrate	UGhent	0.003±0	<0.017	0.01±0	<0.00083	<0.00042
Ammonia water 1	UGhent	<0.0030	<0.017	<0.0022	<0.00083	<0.00042
Pig urine	UGhent	0.02±0	0.02±0	0.03±0	<0.00083	0.001±0
Mineral concentrate	UGhent	0.01±0	0.03±0	0.05±0	<0.00083	0.002±0
Ammonia water 2	UGhent	<0.0030	<0.017	<0.0022	<0.00083	<0.00042
Concentrate after evaporation	UGhent	0.28±0	0.41±0	0.29±0	0.04±0	<0.00042
Liquid fraction of digestate	UGhent	0.01±0	<0.017	0.03±0	<0.00083	<0.00042
Liquid fraction of manure	UGhent	0.01±0	<0.017	0.05±0	<0.00083	0.001±0
Flowering pots made of manure	UGhent	n.d.	n.d.	n.d.	n.d.	n.d.
Keratin-rich foliar spray	UGhent	n.d.	n.d.	n.d.	n.d.	n.d.

n.d. = not determined

Table 10. Mean and standard deviation of Fe, Cu, Zn on dry matter (DM) basis and Cl on fresh weight (FW) basis.

Product code	Project partner	Fe (mg/kg DM)	Cu (mg/kg DM)	Zn (mg/kg DM)	Cl (g/kg FW)
Ash 1	UGhent	34±9.8	15±4.5	1.9±0.96	0.18±0.05
Ash 2	UGhent	<0.0025	<0.0025	2.8±1.2	17±n.a.
Ash 3	UGhent	<0.0025	<0.0025	<0.0025	0.08±0.02
Ash 4	UGhent	<0.0025	<0.0025	1.2±0	42±3.8
Ash 5	UGhent	<0.0025	<0.0025	0.53±0	0.61±0.4
Struvite 1	UGhent	<0.0025	<0.0025	<0.0025	<0.050
Struvite 2	UGhent	<0.0025	<0.0025	<0.0025	<0.050
Compost 1	UGhent	169±78	15±4.5	45±1.6	1.4±1.5
P-poor solid fraction of digestate	UGhent	<0.0025	<0.0025	1.5±0.4	0.07±0.06
Raw digestate	UGhent	205±23	7.3±1.9	31±6.8	2.±0.04
Compost 2	UGhent	5.1±2.4	2.2±0.1	11±0.39	7.5±8.7
Compost 3	UGhent	144±38	12±2.9	51±2.2	2.7±0.06
Compost 4	UGhent	62±13	1.6±0	6.6±2.9	5.4±0.03
Ammonium sulphate	UGhent	<0.00042	<0.00042	5.4±0.34	0.43±0.19
Ammonium nitrate	UGhent	<0.00042	<0.00042	<0.00042	0.04±0
Ammonia water 1	UGhent	<0.00042	<0.00042	0.19±0	0.16±0.02
Pig urine	UGhent	<0.00042	<0.00042	<0.00042	0.03±0
Mineral concentrate	UGhent	<0.00042	<0.00042	0.19±0	0.03±0
Ammonia water 2	UGhent	<0.00042	<0.00042	<0.00042	0.18±0.03
Concentrate after evaporation	UGhent	26±8.4	0.83±0	22±0	6.8±0.19

Liquid fraction of digestate	UGhent	15±0.38	<0.00042	1.1±0.1	0.02±0
Liquid fraction of manure	UGhent	25±1.1	7.6±0.13	13±0.68	0.01±0
Flowering pots made of manure	UGhent	n.d.	n.d.	n.d.	n.d.
Keratin-rich foliar spray	UGhent	n.d.	n.d.	n.d.	n.d.

n.d. = not determined

4. Potential blends

One of the outputs of WPT1 is to propose tailor-made fertilisers – i.e. creating new products with specific nutrient content and ratios through conditioning and blending. Different crops have different NPK requirements in different countries depending on soil types, P and K status of the soil, average crop yield levels and fertiliser recommendations and limitations. In this report, blends are proposed for the most common crops grown in NWE countries. The information about these crops, their nutrient recommendations and NPK ratios has been taken from the report prepared as a part of ReNu2Farm project deliverable in WPT2.D1.2 *'Exploring the demand for recycling-derived nutrients and organic matter in regions of Northwest Europe'*. According to this report, farmland use in NWE is distinguished into grassland, cereal and root crop regions, hence, these three main categories of crops have been considered for blend suggestions. Also, blends for spinach and lettuce were considered, as these are important horticultural crops in Flanders and were used in the ReNu2Farm pot trials.

However, it has to be noted that the indicated crop NPK demand values can only give a rough indication. They were estimated based on agricultural statistics and regional averages. In reality, farming practice differs substantially between single farm enterprises, in particular what concerns access to animal manure as a basis fertiliser as well as soil conditions. Also, management choices as to whether or not to harvest straw influence the crop nutrient need. The estimation takes into consideration factors such as average spring soil N mineralisation. Details on the method on how crop nutrient requirements as well as average manure nutrient input was derived can be consulted in the report WPT2.D1.2. *'Report characterizing farm and crop rotation types in NWE EU and their specific nutrient and organic matter demand'.*

A total of 10 potential blends are proposed in this report, out of which 2 were prepared in the lab and tested in pot trial on spinach and lettuce, and in a soil incubation experiment to test the N release and mineralisation potential of the individual RDFs and their blends. The results of these pot and incubation experiments will be available in WPT2.D2.2

'Report on Nutrient use efficiency determination under controlled conditions' and WPT1.D3.2 *'Report on soil carbon and nitrogen behaviour'*, respectively. The ratio of other 8 blends has been calculated for grass, winter wheat, sugar beet, and potato. The calculations were done considering the NPK ratios of the nutrient demand of the desired crops and NPK ratios of the RDFs. An example is given in Table 11 for Blends 1 and 2 proposed for spinach.

Some proposed blends resulted in higher application dosage of potassium oxide (K_2O) than the recommended dosage of the crops. It was assumed in all these cases that the addition of excess K_2O would not harm those specific crops. Also, research on the micro-nutrient content during blending of RDFs has to be done. Within the framework of this project, performance of blends on plant growth and nutrient uptake was assessed by amending individual RDFs in specific ratios into the soil (for the pot and N-incubation experiments). For future perspectives, it is also essential to perform tests by actual physical blending of RDFs to ascertain possible issue (e.g. reactions such as precipitation, foaming,…). This will help in understanding the changes, if any, in their elemental forms after the formation of the blend. Precipitation or other visible changes during mixing of products should be monitored and pH of the blends formed should be determined. Higher pH values may lead to loss of N in the form of ammonia (NH_3) volatilisation. Maximum care was taken to avoid proposing blends with the RDFs that contained high heavy metal content, especially using ashes, albeit the fact that they were rich in essential plant nutrients like P and K. Further research to study their interactions with plants should be done to observe their suitability as blends.

4.1. Blends for spinach and lettuce

In the second year of the Flemish field trials, spinach will be used as a test crop. Hence, blends were formulated in the laboratory to fit the field nutrient recommendations for spinach (200 kg ha^{-1} N, 55 kg ha^{-1} P_2O_5, 250 kg ha^{-1} K_2O). The following two blends were made and tested in a pot experiment using spinach:

Blend 1 : Ammonium nitrate + Concentrate after evaporation in the ratio 0.5 : 0.5

Blend 2 : Ammonia water 2 + Concentrate after evaporation in the ratio 0.5 : 0.5

Table 11. NPK ratios of the RDFs to be compared with the desired NPK ratio of spinach. The RDFs in the green boxes are the chosen to prepare blend 1 and 2 as they have NPK ratios closest to the required NPK ratio for spinach.

Product code	**N**	**P**	**K**
Ash 1	-	7	1
Ash 2	-	1	2
Ash 3	-	1	2
Ash 4	-	1	5
Ash 5	1	18	1
Struvite 1	1	1	-
Struvite 2	6	10	1
Compost 1	3	1	2
P-poor solid fraction of digestate	21	5	1
Raw digestate	2	1	1
Compost 2	2	1	1
Compost 3	3	1	1
Compost 4	3	1	2
Ammonium sulphate	39	-	-
Ammonium nitrate	82	-	-
Ammonia water 1	107	-	-
Pig urine	2	-	1
Mineral concentrate	1	-	1
Ammonia water 2	155	-	-
Concentrate after evaporation	5	1	10
Liquid fraction of digestate	1	-	1
Liquid fraction of manure	1	-	1
Flowering pots made of manure	NA	NA	NA
Keratin-rich foliar spray	NA	NA	NA
SPINACH	***8***	***1***	***8***

Ammonium nitrate and ammonia water acted as the source of N in each blend, whereas, the concentrate after evaporation provided the P and K along with a minor contribution of N. The blends were calculated considering P as the limiting factor, because Flemish soils have a high content of P. This caused the subsequent blends to be prepared with 15% more K_2O than the recommended dosage of 250 kg ha^{-1}. It was assumed that an over fertilisation with K will not cause any harm to the growth of spinach. However, due to poor germination, the pot trial with spinach failed and the trial was repeated using lettuce as the test crop. The same blends were applied on lettuce at the rate of 200 kg N ha^{-1}, 125 kg P_2O_5 ha^{-1} and 240 kg K_2O ha^{-1}. Here, the blends were prepared considering N as the nutrient of focus, but with P as the limiting factor. This lead to an over-fertilisation of the lettuce with K_2O by 2.2 times than the required dosage. The effect of these blends on lettuce growth is currently being assessed and will be reported in WPT2.D2.2 *'Report on Nutrient use efficiency determination under controlled conditions'.*

4.2. Blends for grass

In the Netherlands, Belgium, Ireland, Northwest UK and regions in France, grassland occupies the largest share of utilised agricultural area (WP T2 D1.2). Two blends are proposed for grass in different locations:

Blend 3 : Ammonium nitrate + Concentrate after evaporation in 0.3 : 1 ratio for silage grass in the Southern region of Ireland on a soil with an average P and K content (weighted average for Southern region, see report WPT2 D1.2 *'Report characterizing farm and crop rotation types in NWE EU and their specific nutrient and organic matter demand'*)

Blend 4 : Ammonium sulphate + Mineral concentrate in 0.5 : 0.5 ratio for mowed grass in the Antwerp region in Belgium on sandy soil

In Ireland and Flanders region of Belgium, large quantities of animal manure are already available for the farmers to apply on the agricultural fields. Therefore, RDFs would only act as a top-up over the average applied or legally allowed limits of animal manure application.

Silage grass in Ireland has a NPK top-up recommendation of 96 kg N ha^{-1}, 25 kg P_2O_5 ha^{-1} and 88 kg K_2O ha^{-1}. Blend 3 fulfils the N and K_2O requirements, but lacks on the P_2O_5 top-up recommendation by 8 kg ha^{-1} In this case, K_2O was considered as the limiting nutrient to theorise the blend because higher levels of K in forages have been known to make cattle more susceptible to diseases like grass tetany, udder edema and milk fever.

The nutrient recommendation for mowed grass in Belgium after animal manure application is 216 kg N_{eff} ha^{-1} and 48 kg K_2O ha^{-1}. No additional P_2O_5 is required in this case as the P limit is fulfilled with animal manure. Unlike the previous case, blend 4 would require an additional K_2O content of 45.5 kg ha^{-1} by synthetic K fertiliser to meet the recommended K dosage. Since the P_2O_5 limit defines the application dosage of top-up fertilisers in this region of Belgium, RDFs rich in K could not be used to prepare blend 4 as this would result in an additional P_2O_5 content which is undesirable as per the recommended dosage. This scenario also indicates the necessity of having higher K concentration in the mineral concentrate.

4.3. Blends for winter wheat

Winter wheat is the basic staple food for human nutrition (bread) in Central Europe. It is also used for livestock fodder. This makes winter wheat a cereal crop grown commonly in the Netherlands, Belgium, France, Germany and the Southeast of the UK. Two blends are proposed for this cereal for 2 locations as below:

Blend 5 : Ammonia water 2+ Concentrate after evaporation in the ratio 0.8 : 1 for winter wheat grown in the Zeeland region of the Netherlands in clayey soil
Blend 6 : Ammonium nitrate + Raw digestate in the ratio 0.4 : 1 for winter wheat grown in the South-West regions of Germany in soil with moderate P and K content

The top-up NPK recommendation in the Netherlands after average manure application is 182 kg N_{eff} ha^{-1} and 17 kg P_2O_5 ha^{-1}. No additional K_2O is recommended for this region, unless the straw is harvested. However, the proposed blend would provide an excess K_2O of 88 kg ha^{-1} as a result of considering P_2O_5 as the limiting factor in proposing the blend.

The over-fertilisation by K_2O is considered to have no harmful effect in the case of cereal growth, but care should be given to balance it out during crop rotation.

Information on crop-specific manure management is lacking for Germany. For the South-West of Germany, manure use is moderately available, with some farmers using a substantial amount, while others do not apply any manure on their fields. However, anaerobic digestion is very common and therefore we suggest here a blend based on digestate. In the case of cereals like winter wheat, it is assumed for this blend scenario that no animal slurry is applied and the straw is not harvested on a soil with moderate P and K content. The nutrient recommendation for such a field is 90 kg N ha^{-1} (for base fertilisation, because for crops like winter wheat, it is common practice to apply a second and third dose of N during the growing season), 64 kg P_2O_5 ha^{-1} and 48 kg K_2O ha^{-1}. The blend is calculated considering the plant-available N present in the digestate, i.e. on the basis of NH_4-N. For an organic RDF like raw digestate, it is also important to consider the organic N (N_{org}) present in the product which will become available by mineralisation within the season of crop growth. The average range of N_{org} in digestate is between 10 - 50% and the blend is calculated considering an average of 30% (and also taking into account 5% loss by NH_4 volatilisation during field application by injection).

In both blend 5 and 6, it is assumed that the excess K_2O will not have any adverse effects on the growth of the cereal crop. However, tests in laboratory and fields need to be performed to validate this hypothesis.

4.4. Blends for sugar beet

Sugar beet is one of the most commonly grown root crop in the NWE regions like the Netherlands, Belgium, France and Germany. It is used for sugar production and was protected by a quota and previously also a fixed price in the EU. Two blends are proposed for sugar beet grown in 2 NWE countries:

Blend 7 : Ammonia water 2+ Concentrate after evaporation in the ratio 0.5 : 0.5 for sugar beet grown in the Netherlands in the Drenthe region with sandy soil

Blend 8 : Ammonium nitrate + Compost 4 in the ratio 0.5 : 1 for sugar beet grown in the Picardie region of France on silty soil

The NPK top-up recommendation for the Drenthe region of Netherlands is 43 kg N ha^{-1}, 13 kg P_2O_5 ha^{-1} and 34 kg K_2O ha^{-1}. Blend 7 meets the top-up requirements. The sensitivity of sugar beet to over fertilisation with K was taken into account before theorising this blend.

Sugar beet grown in Picardie has a recommendation of 130 kg N ha^{-1}, 52 kg P_2O_5 ha^{-1} and 220 kg K_2O ha^{-1}. Here, the N_{org} from compost that would be mineralised during the growing season was also considered before blend proposition. Results from lab measurements show that about 15% N_{org} of compost 4 would be plant-available. Also, blend 8 would require an application of 154 kg K_2O ha^{-1} by synthetic K fertilisers along with RDF application to reach the recommended dosage of 220 kg K_2O ha^{-1}. Here again, the sensitivity of sugar beet to over fertilisation with K was considered before choosing the RDFs to blend.

4.5. Blends for potato

Regions like Northern France, Flanders in Belgium and the Netherlands have a high share of potato cultivation. Two blends are proposed for potato grown in 2 locations:

Blend 9 : Ammonium nitrate + Mineral concentrate in the ratio 0.5 : 0.5 for potato grown in the West Flanders region of Belgium on sandy soil
Blend 10 : Ammonia water 2 + Compost 4 in the ratio 0.5 : 0.5 for potato grown in Picardie in France on silty soil

The top-up nutrient recommendation for West Flanders is 132 kg N ha^{-1} and 147 kg K_2O ha^{-1}. For this blend, an additional application of 145 kg ha^{-1} of synthetic K_2O would be required to achieve the recommended dosage. The limitation on P_2O_5 application hinders the blending of RDFs rich in K_2O because they would bring along P addition into the blends and that is undesirable for Flemish soils. It was also difficult to increase the ratio of mineral concentrate in the blend postulation to meet the K_2O requirements, as this would result in N application of over 50 times than the recommended dosage along with very high volume of RDF application on the field.

The recommended dose of nutrients for the French region of Picardie is 126 kg N ha^{-1}, 52 kg P_2O_5 ha^{-1} and 247 kg K_2O ha^{-1}. Here again, the N_{org} from compost that would be mineralised during the growing season was taken into consideration (15% N_{org} from compost to be plant-available). Also, there is a requirement of additional K_2O application by synthetic fertiliser at the rate of 181 kg ha^{-1} for acquiring the recommended dosage.

An overview of the proposed blends is given in Table 10.

Table 10. Overview of 10 proposed tailor-made blends from the collected RDFs for common crops grown in NWE countries. The recommended dosage for specific crops and dosage from application of proposed blends are stated.

Crop	Regions of NWE where crops are grown	Recommended nutrient dose	Blends proposed	Ratio of blend application
Spinach	Flanders, Belgium	200 kg ha^{-1} N, 55 kg ha^{-1} P_2O_5, 250 kg ha^{-1} K_2O	i) Ammonium nitrate + Concentrate after evaporation ii) Ammonia water 2 + Concentrate after evaporation	0.5 : 0.5
Lettuce	Flanders, Belgium	200 kg ha^{-1} N, 125 kg ha^{-1} P_2O_5, 240 kg ha^{-1} K_2O	i) Ammonium nitrate + Concentrate after evaporation ii) Ammonia water 2 + Concentrate after evaporation	0.5 : 0.5
Silage grass	Southern region of Ireland	96 kg ha^{-1} N, 25 kg ha^{-1} P_2O_5, 88 kg ha^{-1} K_2O	Ammonium nitrate + Concentrate after evaporation	0.3 : 1
Mowed grass	Antwerp, Belgium	216 kg ha^{-1} N, 48 kg ha^{-1} K_2O	Ammonium sulphate + Mineral concentrate	0.5 : 0.5
Winter wheat	Zeeland, the Netherlands	182 kg ha^{-1} N, 17 kg ha^{-1} P_2O_5,	Ammonia water 2 + Concentrate after evaporation	0.8 : 1
Winter wheat	South-West regions, Germany	90 kg ha^{-1} N, 64 kg ha^{-1} P_2O_5, 48 kg ha^{-1} K_2O	Ammonium nitrate + Raw digestate	0.4 : 1
Sugar beet	Drenthe, the Netherlands	43 kg ha^{-1} N, 13 kg ha^{-1} P_2O_5,	Ammonia water 2 + Concentrate after evaporation	0.5 : 0.5

		34 kg ha^{-1} K_2O		
Sugar beet	Picardie, France	130 kg ha^{-1} N, 52 kg ha^{-1} P_2O_5, 220 kg ha^{-1} K_2O	Ammonium nitrate + Compost 4	0.5 : 1
Potato	West Flanders, Belgium	132 kg ha^{-1} N, 147 kg ha^{-1} K_2O	Ammonium nitrate + Mineral concentrate	0.5 : 0.5
Potato	Picardie, France	126 kg ha^{-1} N, 52 kg ha^{-1} P_2O_5, 247 kg ha^{-1} K_2O	Ammonia water + Compost 4	0.5 : 0.5

5. Conclusion

The compositional analysis of products regarding their macro- and micronutrient content together with their plant availability can give an idea about the potential marketability of these products as organic or inorganic fertilisers. Some products such as ammonium nitrate, ammonium sulphate, and ammonia water are entirely mineral N products and do not contain considerable quantities of other macro- and micronutrients (except S in ammonium sulphate). RDFs such as composts have a substantial content of NPK, among other nutrients. In addition, there are products such as ashes produced from various thermochemical processes that are rich in P and K, but they can also contain elevated amounts of heavy metals in them. In general, it was observed between results of different partners, that some RDFs showed higher disparities in the values obtained. These differences could be the result of batches analysed, methods used, and also the inherent nutrient variabilities of the RDFs. These variabilities are clearly visible in the products analysed at various time periods giving a range of results for one parameter (from analyses of Arvalis / AUREA). Another important factor to be considered is the lack of harmonised protocols for assessment of RDFs on an European level, thus causing major incongruities in analyses conducted and subsequently, the results obtained. This emphasises greatly on the need to establish coherent and consistent protocols for not only inorganic synthetic fertilizers (as currently done in Fertilizer Regulation 2003/2003), but also for novel RDFs fertilizers.

After compiling all the data from characterisation, 10 tailor-made fertiliser blends were proposed to suit specific major crops in certain NWE countries based on the crop nutrient recommendation. These recommendations were based on the location, soil type, P and K present in the soil as well as average yield levels and fertiliser recommendations and limits. It was seen that, out of the 24 collected products, blends were mostly made of using ammonium nitrate, ammonia water, pig urine, concentrate after evaporation and, composts made of pig manure slurry after anaerobic digestion and compost from digested household wastes. This is due to the high N content in most of these products coupled with a lower heavy metal load. Another attribute of these RDFs that makes them suitable to be used in blends is their low / negligible P content. The blends with low P content have a potential to be marketed in regions with P-rich soil (e.g. Flanders, the Netherlands). The theorised blends need to be characterised for its physicochemical parameters, and also tested in experiments conducted in controlled and uncontrolled conditions to understand their efficiency as a bio-fertiliser by analysing their agronomic suitability to different crops. Blends 1 and 2 are being currently tested in controlled laboratory experiments to study their effect on the growth of lettuce and the N mineralisation potential. It is also important to observe if the mixing of different RDFs can cause its integral characters to undergo any transition thus causing any negative impacts on the crops.