WPT2, Activity 3

Deliverable 3.1: Protocols for the evaluation of the agronomic value of recycling-derived fertilisers

Authors: Romke Postma and Imke Harms (NMI), Amrita Saju, Caleb Elijah Egene, Erik Meers and Ivona Sigurnjak (U Ghent), Alain Bouthier and Hélène Lagrange (Arvalis Institut du végétal), Tomas Van de Sande and Anke De Dobbelaere (Inagro), Lea Deinert and Achim Schmalenberger (U Limerick), S.M. Ashekuzzaman, C. Redmond, J.B. Murphy and P.J. Forrestal (Teagasc)

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1. Introduction

Within the scope of the Circular Economy a transition will be made to a stronger and more circular economy where resources are used in a more sustainable way. Currently, farmers in countries of Northwest Europe (NWE) mainly use untreated animal manure and mineral fertilisers to fertilise their crops. However, several environmental and ecological problems are associated with this: emissions to the environment, high energy use for mineral nitrogen (N) fertiliser production, reliance on non-renewable mined and imported resources (phosphorous (P) and potassium (K)). At the same time, nutrients end up as waste and are not recycled back to food production: urban wastes such as sewage sludge, food and green waste. For that reason, the Circular Economy should also lead to increasing recycling rates of the plant nutrients nitrogen (N), phosphorus (P) and potassium (K). The NWE Interreg project ReNu2Farm is focused on stimulating the sustainable use of recycling derived fertilisers (RDF's), thus leading to a reduction in the use of non-recycled nutrients in North West Europe.

For a sustainable use of RDF's, the RDF should contribute to crop productivity, soil quality / fertility, while preventing negative effects to water and air quality. For that reason, the following characteristics will be of importance for an evaluation of RDF's concerning their agronomic value:

- The fertiliser value: RDF's containing macro-, meso- or micronutrients contribute to plant nutrition improving crop growth on the short term and maintaining soil fertility on the long term. The total nutrient content, the availability of nutrients for plants and the ratio between nutrients is of importance for the fertiliser value.
- The lime value: this is the capacity of RDF's to increase pH of the soil.
- The organic matter value: the value of the RDF to contribute to the organic matter supply of the soil, which may improve chemical (e.g. N and S mineralization capacity, cation exchange capacity), physical (e.g. soil structure) and biological properties (e.g. disease suppression) of the soil. This may be determined by the organic matter content and the organic matter quality (e.g. stability, C/N ratio, etc.) of the RDF.
- The potential risks for crops, animals, humans and the environment, caused by contaminants, pathogens and other pollutants.

Within this report we focus on the agronomic value, while the environmental impact of RDF's will be described in several other reports of ReNu2Farm. For the characterization of the agronomic value, several methods are used and described in literature. In the following chapters a short overview is given.

2. Characterization of agricultural value

2.1. Characterization of agricultural value of RDF's

Most studies characterizing the agricultural value of RDF's do not describe methodologies for a complete characterization of several agronomic quality criteria but aim to quantify one or several aspects, such as the N and/or P supply (e.g. Velthof et al., 1998; Munoo et al, 2013), of the product. However, in publications that are meant for extension purposes, overviews are given of the fertiliser value of animal manures, composts, biosolids (e.g. sewage sludge) and organic waste materials (e.g. paper crumble) by presenting average nutrient contents and their availability (e.g. Chambers et al., 2013; De Haan & Van Geel, 2014). Next to it, guidelines are supplied for an appropriate use.

In an interesting publication from France, a methodology for the classification and characterization of various organic residues recycled to agriculture has been described, including a field trial method (Bell et al., 2015). The classification is based on the origin of the raw material (urban or industrial organic residue, livestock manures, other animal or vegetal organic residue, mixed organic residue), the type of processing which has been performed (e.g. anaerobic digestion, composting, etc.) and the way of storage. Next to this classification, data about the composition are stored in a database.

2.2. Fertilising substances

As has been stated before, a lot of publications are focused on the determination of one or more aspects of the agricultural value of RDF's. These include the nutritional aspect (supply of nitrogen, phosphorus, potassium, calcium, magnesium and sulphur), the organic matter content and the liming value of the fertilizing products. The relevance and possible methodologies to estimate the agricultural value are listed below:

- **Nitrogen**: a lot of studies have investigated the N supply from various (organic) residues. It is of importance to distinguish between studies that investigated the short term (during (parts of) one growing season) and the long-term N supply (after the first season). The N mineralization from N containing organic residues is still difficult to predict and several indicators have been proposed to be used as a predictor for the N supply (e.g. mineral N content, mineral N as a fraction of total N, C/N-ratio, biochemical composition of the N fraction). Examples of those studies are Cabrera et al. (2005), Velthof et al. (1998), Schröder et al. (2013), Delin et al. (2014), Brockman et al. (2014). Moreover, N losses before, during and after fertiliser application, may affect the N supply by residues.
- **Phosphorus**: the availability of P in residues will be affected by the form of P containing substituents (organic – mineral, calcium-P or iron-P, etc.), but also by the pH in the direct surrounding of the residue after application to the soil. Studies performed are e.g. described by Johnston & Richards (2003), Maguire et al. (2001), Velthof et al. (1998), Kuligowski et al. (2010), Yusiharni et al. (2007) and Delin & Nyberg (2013). In fertiliser recommendations in the Netherlands, UK and Germany, the average P availability of various organic manures has

been indicated (50-100%, see Morel, 2017 for France and Handboek Bodem en Bemesting for the Netherlands).

- **Potassium**: mostly, it is assumed that all potassium that is present in residues is available for plant uptake. For that reason, relatively few studies have investigated the K availability of residues (Möller & Müller, 2012; Ehmann & Lewandowski, 2013). In fertiliser recommendations in the Netherlands, UK, France and Germany, the average K availability of various organic manures has been given (90-100%).
- **Calcium**: calcium is an important constituent of ashes that remains after the burning / incineration of biomass. In wood-ash it is one of the main constituents (e.g. Postma et al., 2011), but different conclusions are drawn about its' availability, varying from low (Arvidsson, 2001; Ring et al., 1999) to high (Demeyer et al., 2001).
- **Magnesium**: magnesium contents in biomass ashes are considerably lower than calcium, and conclusions about its' availability differ also from low (Arvidsson, 2001; Ring et al., 1999) to high (Demeyer et al., 2001).
- **Sulfur**: in organic residues sulphur may partly be present in organic form and partly as inorganic constituents. During some processes, such as anaerobic digestion, sulphur may be lost by mineralization of organic S to sulphate, followed by the reduction of sulphate to H_2S and volatilization (e.g. Möller & Müller, 2012). Sulphur availability from animal manures are generally low. This element is often compared to N assuming its behaviour is similar to N.
- **Liming value**: some residues may have a liming value, which results in a pH increase. This is a well-known property of biomass ashes (e.g. Campbell, 1990; Rechcigl, 1995; Postma et al., 2011; Ehmann & Lewandowski, 2013) and often, its' effectiveness is compared with regular liming materials.
- **Organic Matter**: the application of organic matter with organic residues is often mentioned as its' main purpose. The character of the organic matter (e.g. its' stability, which is the result of the constituents, such as cellulose and lignin) greatly influences its' effect on soil properties, such as aggregate stability and biological activity. In the study of Jérémy et al. (2013) the authors tried to find a relationship between residue characteristics (e.g. fractionation of organic matter in soluble OM, hemicellulose, cellulose and lignin) and its' effect on aggregate stability. However, the fate of organic matter in the soil is determined by a lot of different factors, e.g. besides product characteristics also weather conditions and soil parameters determining the dynamics of soil aggregation. For that reason, a reliable prognosis of organic matter dynamics (e.g. soil organic matter mineralization and integration in soil organic matter) on the basis of biochemical characteristics of organic matter remains challenging and can still be improved.
- **Interaction with soil**: in a lot of situations, the effect of residues on certain soil parameters (e.g. pH, organic matter mineralization, ...), depends not only upon residue properties (e.g. lime value), but also upon the soil properties (e.g. buffering capacity). This interaction should be taken into account during investigations.

2.3.   Types of methods reported

Various types of methods have been reported to study the aspects mentioned above. For the *nutrient supply* of residues, methods described in literature are:

- **Chemical methods**. A first step in the characterisation of the nutrient supply of fertilizing products is the determination of the total contents of nutrients, and sometimes of the various fractions in which the nutrients are present (e.g. organic and mineral N). The total nutrient content is not always the best indicator for nutrient supply. For that reason, a lot of studies were performed to find an indicator, which can be used as a predictor for the nutrient supply from a residue. An example is the amount of organic N that is extracted by a 0,01 M $CaCl_2$ solution, which may be used as an indicator for the N mineralization from that residue (e.g. Velthof et al., 1998).
- **Biological methods**. The supply of nutrients by the biological mineralization process is monitored during a shorter or longer period. This can be done during an incubation experiment, during which the nutrient fractions that are mineralized (e.g. N, P and/or S) are measured frequently. Sometimes, the nutrients are labelled (stable isotopes), to be able to distinguish between several sources of the nutrient fraction studied. In general, these methods are also time consuming, but less expensive than pot and field experiments.
- **Pot experiments and field experiments**. In these types of experiments, the availability of nutrients from the residues is studied by the uptake of those nutrients by a crop. In general, only one nutrient can be studied at the same time (depends on experimental set up). Shortages of other nutrients should be prevented by the supply of sufficient amounts. These types of experiments take a long time and are relatively expensive.
- **Model calculations**. Based on the available knowledge of the biological mineralization process, various models have been developed, which predict the supply of nutrients from a given product, based on the composition of that product and environmental factors. An example of such a model is MINIP, which has been developed for the calculation of the mineralization of N, P and S from various organic materials. For calculations of the N mineralization with MINIP information is required about the decomposability of the organic material studied and the C/N ratio.

For the *characterization of organic matter* in residues, it is of importance to measure its' stability. This can be done with a long-term incubation experiment, in which the organic C decomposition or the CO_2 production is measured. An alternative method is a short-term decomposition, during which the O_2 consumption is indirectly measured by a pressure shift. This method is called the Oxitop-measurement (Veeken et al., 2003). As explained above, prediction of organic matter stability can be done on the basis of biochemical characteristics of organic matter, such as indicator of remaining organic carbon (I_{ROC}) which can be used as an indicator of the humification rate of the organic product (Levavasseur et al., 2020).

For the characterization of the *liming value* of RDF's, several methods are possible. The acidifying or liming effect of mineral RDF's can be quantified by the chemical composition of the product using the so-called Pierre-Sluijsmans formula (Harmsen et al.,1990). In the EU, the liming effect of fertilisers is

characterized by the neutralizing value (NV). For a direct measurement of the neutralizing value of RDF's, one can use titrimetric methods, such as that described by Erich & Ohno (1992). For the determination of the reactivity of liming materials, which includes the dynamics of the liming process, the method of Sauerbeck & Rietz (1985), which has been used for the European standard for testing reactivity (prEN 13971, 2000) of lime fertilisers, has been used. The NV of organic RDFs is not yet well determined (Bouthier et al., 2017), It is important to take into account the N and S cycles in soil, which can impact the acidifying or liming effect. A proton balance should be done to assess the exact acidifying or liming effect but a lot of unknown factors need to be specified.

For the resulting effect of RDF's to the pH of soil, the composition of the soil is also of importance. The pH buffering capacity of the soil determines the amount of lime that is required for a certain pH rise. If the buffering capacity of the soil and the neutralizing value of the RDF is known, the amount of RDF that is required for a certain pH rise can be calculated. The effect on pH can be determined experimentally in incubation experiments with soil (Erstadt et al., 2000; EN 14984).

3. Methods used within ReNu2Farm
3.1. Overview of methods used within ReNu2Farm

Within the scope of ReNu2Farm a set of methods is used to characterize the fertilizing value of RDF's. An overview of the methods used to characterize the N and P fertiliser value and the organic matter (C) supply is given in table 1. These methods are described in more detail in the following chapters and the detailed protocols can be found in the annexes to this report.

Table 1. Overview of methods used in ReNu2Farm to characterize the fertiliser value of RDF's.

Element	Objective	Type	Design	Organisation
N	N mineralization kin.	Lab, incubation	control+reference; duration 5-6 weeks	UGhent
		Field, no crop	control+test; monthly Nmin measurements; duration 12 months	Arvalis
	N fert replace value	Pot	control+ref+test; duration 55 days	UGhent
		Field	control+ref+test; several application levels	Inagro
P	P fert replace value	Pot	control+ref+test; several application levels	Arvalis (contracted Celesta lab)+ UL
		Field	control+ref+test; several application levels	Arvalis + Teagasc
C	Decomposition rate	Lab, incubation	control+reference; duration 120 days	UGhent
C ←→N	CN input vs leaching	Field	balances + Nmin measurements	NMI

3.2. Nitrogen supply

A lot of RDF's are "organic products" containing N (partly) in organic form. As has been stated in the previous chapter, a lot of of studies have investigated the N supply from various (organic) residues. Most of the studies focus on the short-term effect of the N supply during (parts of) the growing season. The N supply of organic RDF's is mostly lower than that of mineral N fertilisers, because of two reasons:

- Not all organic N contained in an organic RDF is coming available for plants by the mineralization process;
- N losses from RDF's before, during and after its' application, esp. by volatilization of NH_3 (ammonia) and denitrification are often higher than those from mineral N fertilisers.

Two types of experiments for the characterization of the N supply may be distinguished:

- Experiments in which the N mineralization kinetics are studies. The mineralization kinetics are of importance for application of organic RDF's in practice, because it gives information about when and how much N is coming available for plant uptake in course of time. These studies may be performed under controlled conditions (lab and/or pot experiments) or in the field. **A protocol for a lab experiment developed in Flanders (by UGhent) is described in Annex 1 and a protocol for a field experiment developed in France (by Arvalis) is described in Annex 2.**
- Experiments focused on the availability of nitrogen from N containing RDF's by the uptake of N by a crop. Mostly, this involves the comparison of the RDF with a conventional mineral N fertiliser and a response curve. From these types of experiments the ANR (Apparent Nitrogen Recovery) and NFRV (Nitrogen Fertiliser Replacement Value) may be derived. It will give us information on the N efficiency of a RDF applied to a specific crop under specific conditions (soil, climate). **A protocol for a pot experiment developed in Flanders (by UGhent) is described in Annex 3 and a field experiment developed in Flanders (by Inagro) is described in Annex 4.**

Within the ReNu2Farm project also RDF's which contain low amounts of organic N fractions have been evaluated (pot trial of Ughent and field trial of Inagro in Belgium). Products such as Ammoniumsulphate and Ammoniumnitrate contain N mainly in a mineral form. In this context studying the aspect of emissions is more interesting to study than the mineralization. Therefore, the incubation and bare-soil field trials as carried out by U Ghent and Arvalis are not relevant for these products but trials to determine ANR and NFRV as done by Inagro in the field and U Ghent in pots indicate the nitrogen fertiliser replacement value of these types of RDF's.

3.3.   Phosphate supply

Phosphate in RDF's and soil is behaving in a different way than N. The availability of P from P containing RDF's may be lower than P in conventional mineral P fertilisers because i) part of the P is present in organic form and should mineralize before it is available for plants and ii) inorganic P is present in a form with a low solubility / availability. However, producers of RDF's are improving their technologies to treat their products in a way that P is more available (e.g. treatment with high temperatures or acids during production, different granule size). It is thus crucial to evaluate the plant availability of the P contained in RDF's.

For that reason, the P fertiliser replacement value (PFRV) of P containing RDF's is often determined in pot and/or field experiments. This was also the case in ReNu2Farm and below the protocols used for the determination are described.

3.3.1. Pot experiments

Within the scope of ReNu2Farm, pot experiments with a crop (ryegrass) are performed in France (Arvalis, subcontracted Celesta lab) and Ireland (University of Limerick) to estimate the short-term P effect of RDF fertilisers. A similar protocol was used, but it showed some differences because of a different focus of the organisations. At the University of Limerick microbial activity for resolubilization of P in soil was studied, whereas at Arvalis the focus was on P uptake to calculate P efficiency.

The protocol of Arvalis is described in Annex 5a and that of the University of Limerick in Annex 5b.

3.3.2. Field experiments

A similar protocol was used to estimate the short-term P effect of RDF fertilisers in field experiments on several locations (in France and Ireland). The protocol showed some small differences because different crops were used as test crop at both locations. Each protocol is written and adapted to its' crops.

- Arvalis chose to work on a cereal, which is a crop in its area of expertise and one of the most representative crops in the North of France. Spring barley has been chosen for two reasons: to match RDF product delivery (which was predicted to be after January) and plant needs and mostly because this cereal is sensible to the lack of P in soils. **This protocol is described in Annex 6a.**
- Teagasc chose to work on grassland which is the most representative crop in Ireland. **This protocol is described in Annex 6b.**

Table 2 compares the key points in both protocols to highlight the differences.

Table 2. Comparison of the differences in the protocol that is used for the short-term effect of P containing RDF fertilisers (PFRV) by Teagasc (Ireland) and Arvalis (France).

	Teagasc (Ireland)	Arvalis (France)
Crop	grassland (Rye Grass)	spring barley
Field	low P status soil (Morgan's P index)	low P status soil (COMIFER tables)
Objectives	Evaluation of the performance and efficiency of RDF in comparison to mineral P (Triple super phosphate)	Evaluation of the performance and efficiency of RDF in comparison to mineral P (Super phosphate)
RDF tested	struvite from potato wastewater	
	struvite from sewage sludge	struvite from sewage sludge (2019)
	poultry litter ash	poultry litter ash (2019)
	sewage sludge ash	sewage sludge ash (2020)
	dairy food processing sludge	
	cattle slurry	
		Compost: solid fraction of pig slurry (30%) and hen droppings (70%) (2019 and 2020)
		Compost: solid fraction of pig slurry (70%) and hen droppings (30%) (2019 and 2020)
		Compost: from solid fraction of pig slurry after digestion (2019 and 2020)
		Sugar beet scum (2019)
		Solid phase of digestate (2020)
RDF amount	40 kg P/ha (recommended dose)	2 doses (recommended dose and half recommended dose)
Control plot	blank	blank
	comparison with mineral fertiliser: TSP (16%): P response curve: 5 doses	comparison with mineral fertiliser: TSP (16%): P response curve: 4 doses
Experimental design	5 rep	3 rep
	plot size: 2m*6m	3m*10m
	trial duration: growing period (February to October)	trial duration: growing period (February to July)
Soil measurement*	initialization: before products application	initialization: before products application
	at harvest: each harvest	at harvest: (one harvest in July)
plant measurements*	for each harvest	for the harvest in July

*the soil and plant measurements are adapted to the practices and laboratory standards of each country.

3.4.   Organic matter (OM) supply

To quantify the organic matter supply by RDF's, it is of importance to know the organic matter content and its' stability. Within ReNu2Farm the stability of organic matter is determined with a long-term incubation experiment under controlled conditions in the laboratory, in which the organic C decomposition or the CO_2 production is measured. **The description of the protocol that is used by the University of Ghent is described in Annex 7.**

3.5.   Nitrogen losses associated with OM supply

Within the scope of soil quality and climate change, the attention for the increase of organic matter contents in soil is receiving a lot of attention. One of the methods to increase organic matter contents in soil, is to increase inputs of organic RDF's. However, this should not lead to adverse effects to the environment, e.g. by an increase in nitrate leaching. For that reason, NMI made a protocol for a field experiment in which the potential nitrate losses that are associated with different organic matter inputs via RDF's are studied**. This is included in Annex 8.**

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Annexes

Annex 1: Protocol N dynamis Lab by U Ghent

Annex 2: Protocol N dynamics Field by Arvalis

Annex 3: Protocol N Fertilizer Replacement Value Pot by U Ghent

Annex 4: Protocol N Fertilizer Replacement Value Field by Inagro

Annex 5a: Protocol P Fertilizer Replacement Value Pot by Arvalis

Annex 5b: Protocol P Fertilizer Replacement Value Pot by U Limerick

Annex 6a: Protocol P Fertilizer Replacement Value Field by Arvalis

Annex 6b: Protocol P Fertilizer Replacement Value Field by Teagasc

Annex 7: Protocol Organic Matter dynamics Lab by U Ghent

Annex 8: Protocol N losses in field by NMI