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**Maa Ja Elintarviketalouden Tutkimuskeskus (MTT)**

MTT Agrifood Research Finland

Revision [1]

## D6.2 Evaluation of the quality, biosecurity and agronomic usefulness of digestates from different digester trials

Lead contractor: **Maa Ja Elintarviketalouden Tutkimuskeskus (MTT)**

MTT Agrifood Research Finland

### Prepared by

Jukka Rintala, Elina Tampio, Tapio Salo (MTT)

Bryan Lewens (AnDigestion)**Contributors**

### *With assistance from*

Filipa Vaz, Constança Correia (Valorsul)

Cristina Cavinato (UNIVE)

David Bolzonella, Cinzia Da Ros (UNIVR)

Ying Jiang, Yue Zhang, Sonia Heaven, Charles Banks (Soton)

Joe Mann, Rebecca Arnold (Greenfinch)

<b>MTT</b>	Maa Ja Elintarviketalouden Tutkimuskeskus, H-Building, Jokioinen, FI-31600, Finland
<b>AnDigestion</b>	AnDigestion Ltd, Biogas Plant Donarbon Site Ely Road, Cambridge, CB25 9PG, UK
<b>Valorsul</b>	Valorização e Tratamento de Resíduos Sólidos das Regiões de Lisboa e do Oeste SA, Plataforma Ribeirinha da CP, Estação de Mercadorias da Bobadela, S. Joao da Talha, 2696-801, Portugal
<b>UNIVE</b>	Dipartimento di Scienze Ambientali, Università Ca' Foscari di Venezia, Calle Larga S. Marta 2137, 30123 Venezia, Italy
<b>UNIVR</b>	Dipartimento Biotecnologie, Università Degli Studi Di Verona, Strada Le Grazie 15, 37134 Verona, Italy
<b>Soton</b>	Faculty of Engineering and the Environment, University of Southampton, Southampton SO17 1BJ, UK
<b>Greenfinch</b>	formerly Greenfinch Ltd (now Biogen), Business Park, Coder Road, Ludlow SY8 1XE, UK

## D6.2 Evaluation of the quality, biosecurity and agronomic usefulness of digestates from different digester trials

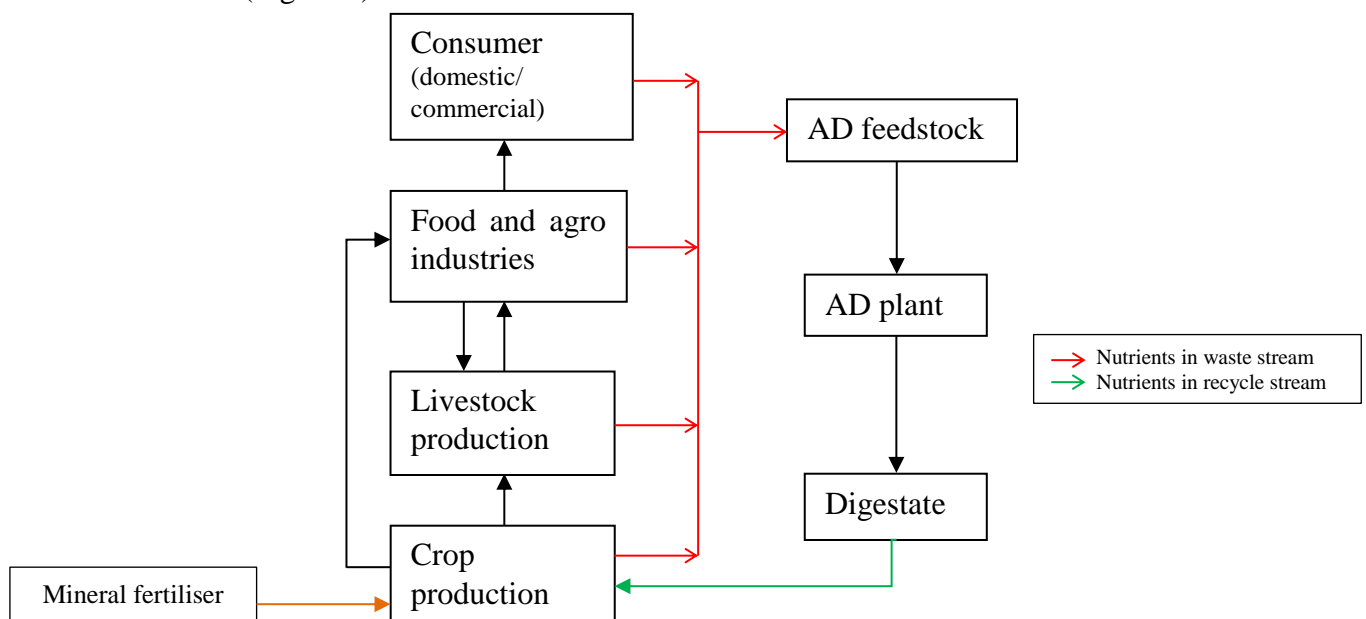
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## D6.2 Evaluation of the quality, biosecurity and agronomic usefulness of digestates from different digester trials

### 1 Introduction

#### 1.1 From food waste to agricultural fertiliser

The depletion of worldwide mineral fertiliser reserves and the fossil fuel use and emissions due to fertiliser manufacturing are increasing the need for sustainable fertiliser production methods. An organic nutrient rich fertiliser can be produced through anaerobic digestion (AD) without fossil fuels and by producing renewable energy at the same time. Anaerobic digestion of various organic materials also enhances the nutrient cycle when the digestate is returned to fields (Figure 1).



**Figure 1.** Digestate and nutrient cycle in organic fertiliser production from food waste digestates (modified from Al Seadi & Lukehurst 2012).

Across the world animal manure is used as a fertiliser due to its nitrogen and phosphorus content, but the raw manure contains low amounts of nutrients easily available to plants, such as ammonium nitrogen ( $\text{NH}_4\text{-N}$ ). If the material is anaerobically digested prior to land application, however, ammonium is formed from the organic nitrogen compounds of the manure and the plant-available soluble  $\text{NH}_4\text{-N}$  concentration will increase (Amon et al. 2006, Clarkson 1990). The anaerobic digestion treatment can also be used for other substrates such as food waste (FW) to produce organic fertilisers and renewable energy in the form of biogas. When food waste collected from households is digested in anaerobic digesters and the digestate is further used as an organic fertiliser in the fields, the nutrient cycle of food production will be closed.

Food waste contains sugars, fibres, fats and proteins of which the proteins are nitrogen-based compounds. During anaerobic digestion organic nitrogen is transformed into water soluble

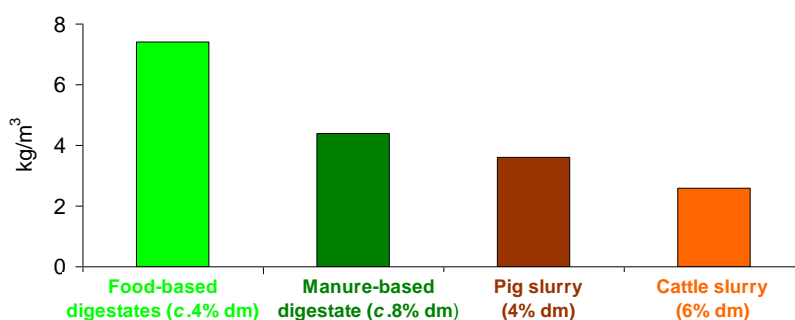
and plant available ammonium and the conversion efficiency depends on the nitrogen concentration and quality of the substrate. Food waste contains also phosphorus, potassium and trace elements, which are essential to plant growth, and all these elements can be found in the digestate after digestion. However, food waste can also potentially contain e.g. heavy metals which may affect the end-use of the digestate. European and national regulations limit the content of PTEs in digestate, and its application to agricultural land, protecting the long term health of soils, and surrounding environments.

## 1.2 Digestate nutrient value

While phosphorus and potassium remain unchanged during anaerobic digestion, the organic nitrogen is degraded and ammonium nitrogen is formed during digestion. The nitrogen and other nutrient content of the digestate are dependent on the nutrient content of the feed, thus the material digested has major effect on the fertiliser quality of the digestate (Haraldsen et al. 2011). When food waste, derived from households and public services (restaurants, canteens, processing plants), is used as feedstock for the digestion process, the amount of plant nutrients is usually high due to the composition of the original waste.

In the soil  $\text{NH}_4\text{-N}$  is converted to nitrate N ( $\text{NO}_3$ ) through nitrification which can lead to N leaching from the soil to water courses or aquifers. To mitigate such losses digestate needs to be applied when crops are growing and can take up the nitrogen, and the amounts applied need to be matched to the crop requirements. In general, typical growth patterns involve high levels of nitrogen take up in the spring and early summer, limited demand in late summer and autumn, and none during winter months when there is no or very little growth. The use of digestate is therefore highly seasonal and as a result significant storage facilities are required.

### Total nitrogen (N) in digestate and livestock slurries ( $\text{kg/m}^3$ )

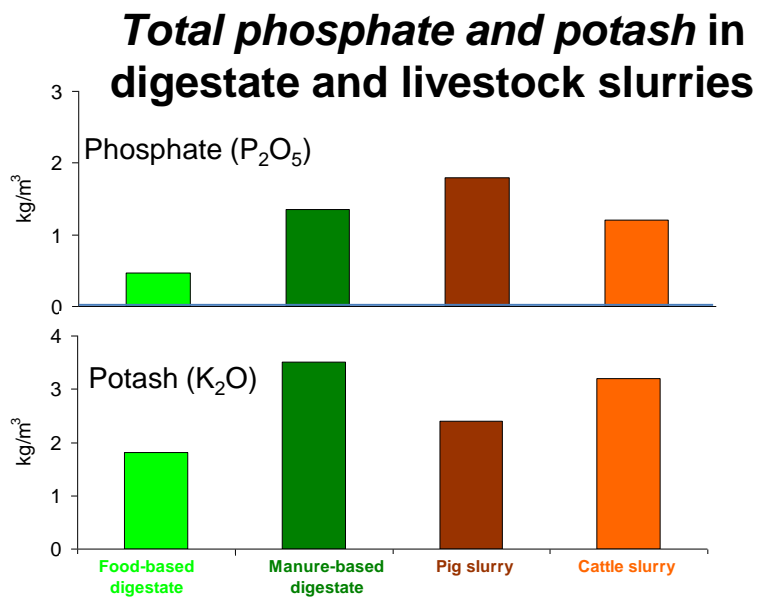


'Typical' slurry values taken from "Fertiliser Manual (RB209)" (courtesy ADAS)

**Figure 2.** Total nitrogen concentration in digestates and slurries.

Figures 2 and 3 give an indication of the variation in nutrient levels between digestates and animal manures. The food-based digestate results were based on the analysis of two Animal By-Product (ABP) compliant plants taking in protein of animal origin which accounts for the

high levels of total nitrogen in the food-based digestate. At the other end of the scale typical cow manure has less than half the nitrogen content of the ABP-compliant AD plants. The proportion of nitrogen in crop available form can be in excess of 80 % total N in food waste digestate, whilst it is typically only about 50 % in cow slurry. The presence of this much available N in food waste based digestate makes it a very useful substitute for mineral nitrogen but the actual analysis will vary depending on input materials, so the agricultural use of digestate needs to be based on the analysis at the time of use. Although the phosphate and potash levels in food based digestate are significantly lower than those found in animal manures (Figure 3) these are present at high enough levels to make a significant contribution to crop up take.



**Figure 3.** Total phosphate and potash concentration in digestates and slurries.

There are small quantities of magnesium and sulphur present in food waste digestate, but the availability of sulphur in digestate is poorly understood; so although digestate application adds sulphur to soil reserves it is difficult to make any claims as to its value in a growing crop.

Use of digestate also adds organic matter to the soils, enhancing the soil fertility; and compared to other organic materials such as animal slurry, digestate is also less decomposable in the soil. Organic matter in slurries has also been reported to decrease the leaching of nitrogen in the soil (Tambone et al. 2010).

### 1.3 Digestate biosecurity

As well as the nutrient value, the biosecurity of the digestate is an essential aspect with respect to the use of the digestate as an agricultural fertiliser. Biosecurity is related to the material pH, organic and dry matter content, physical, inorganic or inorganic impurities and to hygienic quality. Impurities may have effects during the processing to fertiliser products and during fertiliser distribution to the field (physical contaminants); or impurities can weaken the plant growth and soil quality (organic and inorganic contaminants) or affect the after-use of the crop material and animal/human health (organic, inorganic contaminants,

pathogens). Pathogens usually found in FW as a substrate are of different species e.g. *Salmonella*, *Listeria*, *Escherichia Coli*, *Cambylobacter*, *Mycobacteria*, *Clostridia* and *Yersinia*.

Anaerobic digestion acts as a hygienisation process for pathogens depending on the substrate, process temperature and hydraulic retention times (HRT) applied. The effect of process temperature is clear: pathogen inactivation in thermophilic processes (50-55 °C) is calculated in hours but at mesophilic temperatures (30-38 °C) in days (reviewed by Sahlström 2003). Usually when food waste is used as a digester substrate the material also has to be pre- or after-hygenised according to the legislation (70 °C, 1 hour, EU 142/2011). Because the actual concentration of many pathogens in the studied materials can be very low, the hygienic quality of material is tested with indicator bacteria. These indicator bacteria, usually found in great quantities in human and animal intestinal tract, are used to indicate the possible presence of faecal pathogens (Sahlström 2003). Plant pathogens can also be present in food waste and as reported by Termorshuizen et al. (2003) after mesophilic digestion the concentrations of *Ralstonia solanacearum*, (prokaryote, causal agent on potato brown rot) and *Fusarium oxysporum* (fungi, causing wilts and root rots in variety of hosts) were below detection limits. In the same study, however, the inactivation of fungi *Sclerotium cepivorum* (which causes white rot in onions) was not achieved in mesophilic digestion.

Inorganic impurities in the digestate, such as heavy metals, can originate from food production when soils, crops and animal feeds already contain certain levels of heavy metals. During household waste collection heavy metal containing batteries, metal containers etc. can end up in the food waste stream, if not removed during waste pre-treatment steps. During anaerobic digestion heavy metals remain intact and when the digestate is used as a fertiliser they end up in the soil. Heavy metals such as nickel, zinc, copper and chromium are also considered as trace elements and vital for animal and human health in small concentrations. If the concentration increases over the threshold value, however, these heavy metals are considered harmful for health and environment (Al Seadi & Lukehurst 2012).

Organic impurities such as PAH- and PCB-compounds, dioxins and furans (PCDD, PCDD/F) are persistent organic pollutants (POPs) which are only sparingly biodegradable in the natural environment. These compounds originate e.g. from industrial processes and are usually connected with the treatment of wastewater, but organic pollutants can be found also in food wastes and further in food waste digestates. Other organic contaminants present in digestates are halogenated organic compounds (AOX, adsorbable organically bound halogens), LAS (linear alkylbenzene sulphonate), DEHP (di(2-ethylhexyl) phthalate) and NPEs (nonylphenols). Physical contaminants such as plastic and metal pieces or large organic pieces that cannot be digested in the process can affect the end use of digestate by causing problems in the spreading machinery and uneven fertiliser application.

## 1.4 Digestate use

### 1.4.1 Pre-treatment

Digestate originating from organic waste digestion can often be used as a fertiliser as such without any pre-treatment. Digestate processing methods can be divided into processes where the nutrient concentration of the material is increased compared to the original digestate, or where the aim is to produce separate nutrient-containing mineral fertiliser-like materials.

Because the water content of the digestate is usually quite high and transportation of the high water content digestate is inefficient, the first processing method is to increase the solids content (usually to >25 %, Table 1). To achieve this, digestate can be processed by similar methods as used in the wastewater treatment industry for treatment of sludges, e.g separating the solid and liquid fractions by a screw press, belt press or decanter centrifuge. In these technologies the liquid fraction contains the soluble nitrogen and potassium of the digestate while phosphorus remains in the solid fraction. The solid fraction can be also further dried, pelletised or composted to increase transportability and marketing value (Lukehurst et al. 2010).

Thermal treatments can also be used to evaporate water from the digestate by using surplus energy from combustion of the biogas produced from digestion, external energy or solar energy. Ammonia volatilisation may be a problem during the drying process. Volatilisation can be prevented by changing the pH of the material, but this can lead to foaming problems. Other methods to produce nitrogen-rich liquid fertilisers (mineral fertiliser-like products) from digestate are membrane technologies (nano- and ultrafiltration) incorporated with reverse-osmosis; ammonia stripping (see also VALORGAS deliverable D3.6); struvite precipitation (see also VALORGAS deliverable D4.7); and ion exchange.

**Table 1.** Nutrient concentrations in separated source-segregated food waste (ss-FW) digestate, mechanically-recovered organic fraction of municipal solid waste (mr-OFMSW) digestate and composted FW digestate.

	Unit	Liquid fraction <sup>a</sup>		Solid fraction <sup>a</sup>		Compost <sup>b</sup>
		ss-FW	mr-OFMSW	ss-FW	mr-OFMSW	
TS	% FM	5.84	6.57	14.7	35.0	N/A
Total-N	kg tonne <sup>-1</sup> FM	112	48.1	54.7	16.2	9.0-28.0
Soluble-N	kg tonne <sup>-1</sup> TS	65.1	22.4	23.6	4.76	N/A
Total-P	kg tonne <sup>-1</sup> TS	11.9	4.52	10.5	3.4	1.8-9.3
Total-K	kg tonne <sup>-1</sup> TS	46.1	17.5	18	3.89	3.4-23.0
Total-C	kg tonne <sup>-1</sup> TS	N/A	N/A	N/A	N/A	191-470

<sup>a</sup>Values adopted from Zhang et al. 2012

<sup>b</sup> Values reviewed in Boldrin et al. 2009

## 1.4.2 Composting

Anaerobic digestate can also be composted after digestion to produce a compost-like fertiliser/soil conditioner. Usually composting is applied to the separated solid fraction of the digestate. The composting process also functions as a hygienisation process, if the temperature and retention time are high and long enough. Because composting is an aerobic process bulking material is used to keep the total solids high enough to ensure aeration through the compost pile. Also forced aeration and different composting techniques (e.g. pile, tunnel or drum composting) can be applied.

Composting increases the solids content and also the carbon and nitrogen content of the material depending on the composition of the bulking material (Table 2): the solids content can increase to over 70 % (Tambone et al. 2010). Compared to digestate the nutrient content in compost is lower, but the organic matter content is high, which enhances the soil



amendment capacity of the compost (Tambone et al. 2007). By composting also the phytotoxicity of the FW digestate has been reported to decrease (Abdullahi et al. 2008). In Portugal, composting of anaerobic digestates is currently the only way to produce organic fertiliser material from food waste digestate. At Valorsul, Portugal, the food waste digestate is first dewatered and then composted in a composting tunnel with forced aeration. After one week the composting is continued in windrows and finally refined by sieving (Vaz 2013).

### 1.4.3 Storage

After anaerobic digestion of food waste the digestate has to be stored properly before use. Storage is essential because the application time of fertilisers according to European legislation is restricted to cover only the growth season of plants (EEC 676/91). Since AD plants operate with a steady supply of input materials, digestate storage is a key factor in best practice in the use of digestate as a fertiliser replacer. Grass is useful crop for digestate application since frequent harvests by cutting or even grazing lengthen the spreading season. Grazing can, however, be more problematic due to the 21-day grazing ban (applied in the UK and in Finland). In the UK, in average years a storage period of 6-7 months production should be sufficient in a grassland area although if the very wet year 2012 is an indicator of more extreme weather patterns ahead, even this may not be enough. In all arable areas the spreading season can be even more concentrated, as crop nitrogen demand for all the main arable crops is in the spring and early summer. There are some crops such as autumn-sown oil seed rape where there is a limited requirement for nitrogen later in the year, but the application rates are much lower. Arable farming tend to be in areas with lower rainfall so early spreading opportunities are much greater. Storage requirements in these areas could be up to 9-10 months. In Finland, nitrogen-containing fertilisers cannot be spread between 15 October - 15 April, which means a minimum of 6 months' storage. If the soil is not frozen and is dry in spring, the fertiliser can be applied by 1 April and in autumn until 15 November. For grass crops nitrogen fertilisers can be applied only until 15 September.

Digestate should be stored in a manner to prevent gaseous emissions and nutrient losses from the material. Due to nitrogen and carbon degradation, compounds such as ammonia ( $\text{NH}_3$ ), nitrous oxide ( $\text{N}_2\text{O}$ ) and methane ( $\text{CH}_4$ ) are formed. The levels of produced gaseous emissions during storage are dependent on the temperature, dry matter content, nitrogen content, pH, and storage conditions (cover material, surface area) of the digestate (Amon et al. 2006, Rehn & Müller 2011). Nitrous oxide is formed through ammonia nitrification and nitrate de-nitrification processes in aerobic conditions (Amon et al. 2006), and ammonia is formed through volatilisation of ammonium ( $\text{NH}_4\text{-N}$ ,  $\text{NH}_4^+$ ). The rate of  $\text{NH}_3$  formation is highly dependent on the ammonium concentration of the digestate and the volatilisation can greatly decrease the  $\text{NH}_4\text{-N}$  content of the digestate. Residual methane produced in anaerobic conditions during the digestate storage, however, is a harmful greenhouse gas (GHG) if released to atmosphere (Rehl & Müller 2011).

The digestate can be stored in covered storage tanks, lagoons or bags to prevent ammonia losses to the atmosphere and also residual methane losses from the material. The cover material of the storage facility can be a membrane, concrete, steel or a floating cover of straw, clay granules or plastic on top of the liquid surface. If the cover of the digestate storage is air tight the residual methane potential can be utilised and the GHG emissions during storage decreased (Clemens et al. 2006). Also  $\text{N}_2\text{O}$  emissions can be prevented by covering the material in a gas-tight manner and storing the digestate in anaerobic conditions. By this

technique 90 % of the gaseous emissions ( $\text{CH}_4$ ,  $\text{N}_2\text{O}$ ,  $\text{NH}_3$ ) can be reduced and 55-100 % of  $\text{NH}_4\text{-N}$  retained in the digestate (Rehl & Müller 2011).

Storage is expensive, however, and can easily be a limiting factor on digestate utilisation, so usually all spreading opportunities are taken that will not compromise the environment. This can be a difficult distinction when there are limited storage facilities and an AD plant that is a continuous process. Retrofitting covers on existing farm stores is expensive and frequently not practical and new stores fitted with covers are costly in relation to open-topped stores.

There is now interest in covered lagoons as a lower cost option, such as the example shown in Figure 4. The lagoons are fitted with a plastic cover supported by floats. Rain water is channelled to a collection point where it can be pumped away as clean water.



**Figure 4.** Lagoon fitted with floating cover.

#### **1.4.4 Application to land**

As during the storage stage of digestate utilisation so also during land application, gaseous compounds ( $\text{NH}_3$ ,  $\text{CH}_4$ ,  $\text{N}_2\text{O}$ ) can be released to the atmosphere, decreasing the digestate ammonium content and increasing GHG emissions. Volatilisation rates are sensitive to dry matter content and, when applied to a soil which is reasonably dry, digestate should percolate into the soil quickly resulting in reduced emissions. Conversely ammonia emissions increase with pH and ammonia is particularly prone to volatilisation at typical pH levels for digestate.

There has been a considerable amount of work done on estimating losses through volatilisation, and in a review carried out by Lalor et al. (2012) losses of over 70 % of total  $\text{NH}_4\text{-N}$  (TAN) from slurry have been reported following splash plate application. The extent of volatilisation is dependent on atmospheric and soil conditions at the time of spreading and is also heavily influenced by the pH and dry matter of the material being spread. Volatilisation increases in warm, dry and windy conditions and during application to wet

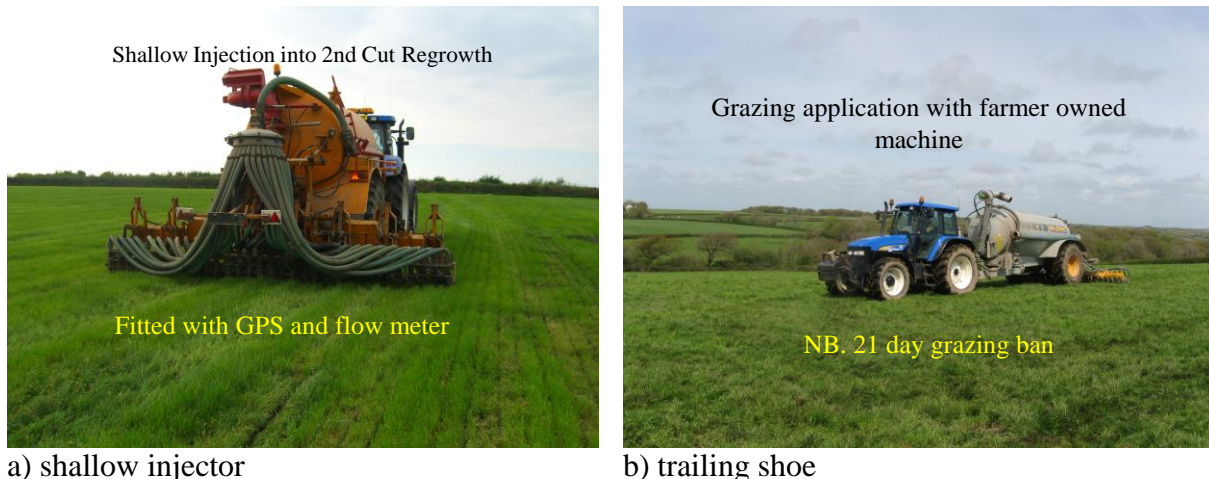
soils. Digestate can have three to four times the crop available nitrogen content of slurry, so it is also important that application rates are accurate and precise. Care therefore has to be taken in choosing the spreading technique and in assessing application rates to meet crop nitrogen requirements.

The application technique and the pre-treatment of the digestate affect the material losses during application. Slurry-like digestates can be spread with same equipment as slurries; with splash plate, trailing hose, trailing-shoe or injection (Lukehurst et al. 2010). More solid materials can be spread to the fields with compost or manure spreaders. To ensure even and accurate spreading of digestate and to reduce ammonia volatilisation during spreading techniques such as trailing hose, trailing-shoe and injection should be used (Table 2). In all these techniques the digestate is spread directly on or under the soil surface while the splash plate spreads the digestate to the air increasing the volatilisation and also other environmental risks. For this reason the use of splash plates is banned in some countries (Lukehurst et al. 2010). With the trailing-shoe and injection techniques the digestate is applied under the top soil which also minimises the risk of odours.

**Table 2.** Characteristics of digestate spreading methods (adopted from Birkmose 2009).

	Splash plate	Trailing hose	Trailing shoe	Injection
Distribution of slurry	Very uneven	Even	Even	Even
Risk of ammonia volatilisation	High	Medium	Low	Low or none
Risk of contamination of crop	High	Low	Low	Very low
Risk of wind drift	High	Minimal after application	Minimal after application	No risk
Risk of smell	High	Medium	Low	Very low
Spreading capacity	High	High	Low	Low
Working width	6-10 m	12-28 m	6-12 m	6-12 m
Mechanical damage on crop	None	None	None	High
Cost of application	Low	Medium	Medium	High
Amount of slurry visible	Most	Some	Some	Very little

In Figure 5 the shallow injection tanker is fitted with a flow meter, and with a skilled operator good spreading precision can be achieved (actual spread rate variation from planned spread rate <5 %). Applying digestate with such equipment can typically cost £4.50 m<sup>-3</sup> (5.2 € m<sup>-3</sup>) in UK conditions (Nix 2013, ADAS personal communication). Using equivalent ADAS/ Nix figures shallow injection spreading by trailing hose/shoe would typically cost around £4 m<sup>-3</sup> (4.6 € m<sup>-3</sup>) and broadcast/splash plate £3 m<sup>-3</sup> (3.5 € m<sup>-3</sup>). Umbilical spreading costs can range considerably as a lot of time is involved in setting up and moving pipework between fields, but where large amounts of digestate are to be spread over significant areas very high spreading rates can be achieved thus reducing the unit spreading costs.



a) shallow injector

b) trailing shoe

**Figure 5.** Different spreading options for digestates: (left), (right).

## 1.5 Legislation concerning digestate use in agriculture

### 1.5.1 EU

Digestate and digestate-derived compost use in all of the VALORGAS partner countries (UK, Finland, Italy and Portugal) is regulated by EU legislation. The most significant regulations are those concerning animal by-products (EC 1096/2009, EU 142/2011), fertilisers (EC 2003/2003) and the protection of waters against pollution caused by nitrates from agricultural sources (676/01/EEC). The Animal By-product regulations (EC 1096/2009) provide three categories for materials according to the risk to public health and animal health, and regulate the disposal and use of those materials. Materials from category 3, such as food and catering waste, can be treated in biogas plants after hygienisation (70 °C, 1 hour) and the digestate can be used as a fertiliser. Category 2 materials (e.g. *Salmonella* contaminated slaughterhouse materials), however, must be pressure sterilised (>133 °C, 20 min) before digestion. According to the regulation the digestion residue can be placed on the market and used as organic fertiliser or soil improver. The quality of the digestion residue must meet hygienic standards; the threshold values for *E. coli* or Enterococcaceae are 1000 cfu g<sup>-1</sup> when no *Salmonella* is detected in 25 g sample (EU 142/2011).

The fertiliser regulation (EC 2003/2003) is related to inorganic fertilisers. The regulation includes inorganic primary, secondary and micro-nutrient fertilisers, their fertiliser types, marking, labelling and packing. The EU's nitrate regulation (EEC 676/91) aims to protect waters against pollution caused by agricultural nitrates. The regulation sets measures for national action programs concerning nitrogen fertiliser use e.g. soil and climate conditions, plant requirements. According to the regulation the allowed amount of nitrogen that can be applied in manure is 170 kg N ha<sup>-1</sup> year<sup>-1</sup>.

### 1.5.2 Finland

In Finland digestate use in agriculture is regulated by the Law on fertilisers (539/2006) and the decree of the Ministry of Agriculture and Forestry (24/11) which together implement the EU's Animal By-product and fertiliser regulations. The decree (24/11) defines type names for fertilisers and soil amendments and sets minimum values for nutrients and also limits for contaminants such as heavy metals (Table 3), pathogens and plant pathogens in inorganic and

organic fertilisers and soil amendments. Limits for pathogens in the decree are obtained from the EU regulation (EU 142/201). For soil amendments the decree defines also limits for the stability of the material ( $3 \text{ mg CO}_2\text{-C g}^{-1} \text{ VS day}^{-1}$  for packed materials).

In Finland, the limit in the EU's Nitrate Directive (EEC 676/01) for total nitrogen in manure ( $170 \text{ kg N ha}^{-1} \text{ year}^{-1}$ ) is also considered as the limiting factor for other organic fertilisers such as digestate. Limits for soluble nitrogen and phosphorus, however, are controlled by the Finnish agri-environmental aid program (MAVI 2009) which sets soluble-N limits for a variety of crops with respect to different soil types which are typical in Finland. The soil types are also divided between northern and southern Finland. The phosphorus fertilisation control, however, only takes into consideration the crop type and the soil fertility (7 classes). According to the environmental-aid program, 100 % of the soluble-P content is taken into consideration if the organic fertiliser originated from animals. If the fertiliser contains materials (sludges) from wastewater treatment, 40 % of the total-P content is taken into consideration.

**Table 3.** Limits for inorganic and organic contaminants in fertilisers according to the Finnish and English legislation and Portuguese proposed legislation.

	Finland		Portugal			UK
			Class I	Class II	Class IIA	
Heavy metals ( $\text{mg kg}^{-1}$ TS)						
As	25	N/A	N/A	N/A	N/A	N/A
Hg	1	0.7	1.5	3	5	1
Cd	1.5	0.7	1.5	3	5	1.5
Cr	300	100	150	300	400	100
Cu	600	100	200	400	600	200
Pb	100	100	150	300	500	200
Ni	100	50	100	200	200	50
Zn	1500	200	500	1000	1500	400
Organic contaminants ( $\text{mg kg}^{-1}$ TS TS)						
AOX	N/A			500		N/A
LAS	N/A			2600		N/A
DEHP	N/A			100		N/A
NPE	N/A			50		N/A
PAH	N/A			60		N/A
PCB	N/A			0.8		N/A
PCDD and PCDF	N/A			100 <sup>a</sup>		N/A
Other (%TS)						
Physical contaminants	<0.5 <sup>b</sup>	0.5	1	2	3	0.5
Stones	N/A	5	5	5	N/A	8

<sup>a</sup>  $\text{ng TE kg}^{-1}$  TS

<sup>b</sup> % FM, including stones

N/A= not available

### 1.5.3 Italy

In Italy, according to D.lgs 152/2006 and 205/2010 (deriving from 2008/98/EC, Waste Framework Directive) the application of digestate as produced for agriculture purposes is not allowed since digestate, even if it undergoes biological processing, is still a waste. The only way to upgrade digestate from waste to product status is to compost it together with green waste, in order to create a fertiliser. In these conditions, digestate is converted into a product and is then considered under the Fertiliser legislation (D.lgs 75/2010).

On the other hand digestate produced by the treatment of livestock effluents and energy crops or wastewater sludge can be used directly for agricultural purposes provided it meets the specific regulations on nitrogen loadings in Nitrate Vulnerable Zones (NVZ) and on heavy metals and pathogen content.

### 1.5.4 UK

In the UK regulation plays a major role in the way digestate is used and indeed in the way that the AD industry is structured. As a result of devolution, regulation in England and Wales varies from that in Scotland and Northern Ireland although the various regulations are all compliant with the relevant overlying European Directives. For the purposes of this report only regulation in England and Wales is considered.

There are Europe-wide concerns over excess nitrates in fresh water and in England it is estimated that 50-60 % of nitrates arise from agriculture. England has designated just under 60 % of its area as NVZs in order to comply with the European Nitrates Directive (EEC 676/91), with a view to controlling the nitrate levels in the water. Farms within NVZs are legally obliged to obey a number of rules covering the use of manures and other organic materials including digestate. NVZ regulations build on the Code of Good Agricultural Practice (COGAP) (Defra 2009) and are part of the cross-compliance set of requirements that farmers must meet in order to get their Single Farm Payment (EU area-based payment scheme). Failure to meet these requirements can result in part or all of the payment being forfeited.

The most important NVZ regulations relevant to digestate are as follows:

- **Livestock manure N farm limit:** A loading limit of 170 kg ha<sup>-1</sup> of total N from livestock manures (deposited during grazing and by spreading) per calendar year, averaged across the farmed area. This is calculated by using standard nitrogen production figures for animals on the holding throughout the year. It is only relevant to digestate if any of the input materials originate from livestock manures. Many food waste digestion plants in the UK do not take in manures, in which case this limit does not apply. If any manures are used then an apportionment must be made, or all the inputs can be categorised as animal manures. There is a clear incentive for operators not to include animal manures if they are supplying farms in NVZs.
- **Organic manure N field limit:** A spreading limit of 250 kg ha<sup>-1</sup> of total N establishes a maximum application rate for organic manures – this is based on a rolling year.
- **Closed period (organic manures):** This prohibits the spreading of organic manures with high available nitrogen content during specified periods. This includes materials with an ammonium N content greater than 30% of total N and therefore includes digestates. Storage during the closed period is therefore a necessity.

- **N Max limit:** This is a limit of the amount of mineral nitrogen and crop available nitrogen that can be applied to individual crops in any one year. At present digestate from non animal manure sources is not covered by this rule but will be included when the regulations are changed in 2013.
- **Record Keeping:** A number of records have to be kept by the farmer in order to demonstrate cross-compliance. The use of digestate is included in those records. There are comprehensive software packages available that cover these requirements.

It is becoming noticeable that on some highly stocked farms soil phosphate levels are rising, and although food waste digestate generally has low phosphate levels it can be difficult to justify the application of digestate in some cases. The Environmental Agency (EA) expects farmers to manage their soils to achieve a soil index of 2 and will raise concerns at higher levels while at very high levels (Index 5) no application at all should take place.

It has been possible since 2010 to supply digestate as a product. The Department for Environment, Food and Rural Affairs (Defra) funded the development of the PAS110 standard and a Quality Protocol which sets out the criteria for the production and use of digestate as a quality product (BSI, 2010). This was developed by the EA and the Waste and Resources Action Programme (WRAP) in consultation with interested parties including retailers, processors, operators and regulators. Currently seven UK plants are registered as compliant on the Biofertiliser Certification Scheme website. Digestate from AD plants processing food waste that is PAS110/QP compliant will have a pasteurising stage in place and the limits for the hygienic quality are the same as in the EU legislation. The heavy metal limits for digestates are the same as those specified for compost in PAS100 (BSI, 2011) and are set in mg kg<sup>-1</sup> dry matter (Table 4). The actual heavy metal amounts applied to land are very small indeed since annual applications are usually limited either by crop demand for nitrogen or the COGAP limit of 250 kg N ha<sup>-1</sup> year<sup>-1</sup>.

As an example of digestate utilisation an AD plant in Holsworthy, UK, is introduced. The Holsworthy plant has been in operation for over 10 years, and was originally designed to process mainly animal manures with some additional food waste. Pasteurisation was installed at the time of construction, not to meet ABP regulations (which were not introduced until 2003). but to address farmer fears of cross contamination of bovine tuberculosis via the cattle slurry. Since 2008 only food wastes have been processed through the plant to meet all ABP regulations.

Table 4 gives an analysis of the key nutrient qualities of whole digestate produced at the Holsworthy. Note the high pH levels, low dry matter and high proportion of potentially crop available nitrogen. Holsworthy digestate is potentially very attractive to farmers because, having high levels of ammonium N, it can be used as a replacement for expensive industrially made mineral nitrogen. Nitrogen in this form has to be carefully managed, however, if it is to be used by the crop and not lost to the environment where it has the capability to cause harm.

Some AD plants in the UK and elsewhere have their own land on which they can spread digestate. Compromises can be made on crops to be grown and the proportion of nutrients from digestate to be used. Holsworthy does have some land of its own but the major proportion of the digestate is exported to other farmers. The plant has been operating for over 10 years, so local farmers are in general aware of the properties of digestate and reassured

over the safety of using it. The key promotional tool used is word of mouth between farmers. This works for both good and bad, so if any issues are raised by farmers they need to be dealt with quickly to avoid adverse comment circulating.

**Table 4.** Holsworthy digestate analysis. Key constituents.

Analysis	Value	Unit
pH	8.4	-
Dry matter	4.6	%
Total nitrogen*	6.3	kg m <sup>-3</sup>
Ammonium nitrogen	5.9	kg m <sup>-3</sup>
Phosphate	1.1	kg m <sup>-3</sup> P <sub>2</sub> O <sub>5</sub>
Potash	1.4	kg m <sup>-3</sup> K <sub>2</sub> O
Magnesium	0.1	kg m <sup>-3</sup> MgO
Sulphur	0.9	kg m <sup>-3</sup> SO <sub>3</sub>

\*12 month COGAP application limit 39 m<sup>3</sup> ha<sup>-1</sup> (250 kg N ha<sup>-1</sup>)

Farmers use digestate as a means of saving money, and the key nutrient they consider is nitrogen. Digestate is high in available nitrogen and it is the nutrient that when applied has the greatest visible effect. To comply with both product and 'waste' status rules, recent soil samples for the land to be spread must be available so that nutrient recommendations can be made. On the grassland farms that are prevalent around Holsworthy few farmers were regularly soil testing their land. Soil tests are now routinely carried out as part of the Andigestion digestate supply service. It is only then that any issues surrounding phosphate and potash issues are realised. Although rare in the Holsworthy area, farmers with significant arable areas are likely to be more up to date with soil sampling.

Farmers also want digestate applied at the correct time. Near Holsworthy large areas of grassland are closed off for silage in the early spring, and digestate will be required for spreading as soon as the ground is dry enough. Ground temperature is rarely a limiting factor in this area. Further applications can be applied for further cuts later in the growing season. Very little digestate is applied on winter cereals in this area since there is no crop requirement for nitrogen in the autumn when the crops are sown, and opportunities to spread in the spring on growing crops are limited because ground conditions are rarely suitable due to high rainfall and heavy soils. The demand for digestate is therefore highly concentrated into the spring and early summer months.

Table 5 gives an example of putting a value on digestate using nutrient values for mineral fertilisers, but the value will vary depending on the basis of the valuation. The nutrient values used are based on the current fertiliser prices from a local merchant for nitrogen, phosphate and potash (UK). The value of a cubic metre (1 tonne equivalent) is relatively low at £6.9 m<sup>-3</sup> and since only the ammonium fraction of the nitrogen is potentially available to the crop a further deduction is made to allow for this in (B).



**Table 5.** Digestate calculation example.

<b>Digestate Recommendation and Valuation</b>					
Digestate spread on grassland in the Holsworthy area.					
<i>1<sup>st</sup> Cut Silage</i>		<b>Spreading Rate (tonnes ha<sup>-1</sup>) = 24</b>			
<i>Digestate spread shallow injection March 24<sup>th</sup></i>		Nitrogen (N)	Phosphate (P <sub>2</sub> O <sub>5</sub> )	Potash (K <sub>2</sub> O)	Value
A	Estimated total nutrients in digestate (kg m <sup>-3</sup> )	6.3	1.1	1.4	£6.90
B	Estimated available nutrients in digestate (kg m <sup>-3</sup> ) <sup>a</sup>	5.9	1.1	1.4	£6.55
C	Requirement for 1 <sup>st</sup> cut silage (kg ha <sup>-1</sup> ) <sup>b</sup>	120	40	80	£174.87
D	Calculate digestate supply of potentially plant-available nutrients (kg ha <sup>-1</sup> )	142	26	34	£157.24
E	Calculate supply of plant-available N after NH <sub>3</sub> and NO <sub>3</sub> losses (MANNER-NPK <sup>c</sup> ) (kg ha <sup>-1</sup> )	120	26	34	£138.64
F	Balance required	0.0	14	46	£36.23
G	Total N applied per hectare (kg ha <sup>-1</sup> )	151			

a. Ammonium N content.  
 b. Derived from RB209  
 c. MANNER- NPK  
 Soil Index P= 2 and K= 2-

<i>Fertiliser Prices as at 1<sup>st</sup> Jan 2013</i>	%	Price tonne <sup>-1</sup>	Price kg <sup>-1</sup>
Ammonium Nitrate	34.5	£297	£0.86
Muriate of Potash	60.0	£330	£0.55
Triple Super Phosphate	46.0	£285	£0.62

The example given is for a first cut of grass silage yielding 23 tonnes ha<sup>-1</sup> at 25 % dry matter. Nutrient requirements have been derived from the Fertiliser Manual (Defra 2010) which is a government-backed industry standard. The value of the nutrients removed is considerable (D) at nearly £160 ha<sup>-1</sup>.

In the past there has been no specific published guidance on how to assess nitrogen losses from leaching or volatilisation when spreading digestate, although losses could be assessed using a freely available programme called MANNER (ADAS 2010). No specific data was available on digestate but reasonable calculations could be made based on pig slurry. At the time of writing a revised version has been released by ADAS called MANNER- NPK (ADAS 2010) which for the first time includes figures for food based digestate. The software allows the calculation of crop available nitrogen to be made based on a number of variables including crop, soil type, application rates, dry matter, rainfall, method of application and soil and atmospheric conditions at the time of application.

In the example given (E) the digestate is being spread when the crop is starting to grow and the soil is drying out, so leaching losses are not expected. The estimated losses are from volatilisation and some de-nitrification. Very small amounts of nitrogen are calculated to become available for a subsequent crop in the same season and crops in the following season. The application rate chosen for the example provides all the nitrogen requirements for that

crop and demonstrates a shortfall on the crop maintenance requirement for phosphate and potash.

After allowing for nitrogen losses, but including the small amount of nitrogen that will become available for subsequent crops, the digestate applied is worth £140 ha<sup>-1</sup>. Despite the losses the application provides nearly 90% by value of the estimated crop off take but the value of one cubic metre of digestate is only £5.90. The significant costs of managing digestate before it is applied to the soil can thus easily offset the value of the digestate.

### 1.5.5 Portugal

In Portugal the digestate from anaerobic digestion is not applied to land because of lack of legislation concerning digestate use. Prior to application the digestate is first treated in aerobic composting process. There is, however, a proposal for legislation concerning organic fertiliser use in agriculture in Portugal which sets limits for digestate-derived compost applied to soils. In this report the limit values used are from the proposed fertiliser legislation (MEID 2010). According to the proposal, organic fertilisers are divided into 4 classes (I, II, IIA, and III). Limits for heavy metals, organic and inorganic contaminants (stones and other inert material) contaminants and hygiene indicators are defined for all classes. The fertilisers are divided into classes according to their heavy metal concentrations (Table 3).

The proposal for organic fertiliser use in agriculture defines that fertiliser from classes I and II can be used in agriculture, while materials from class IIA are suitable also for wine, fruit and olive farms as well as for landscaping and gardening. The annual heavy metal quantity spread to soils is also defined in the proposal and also the quantity of the fertiliser material.

### 1.6 Study of digestate samples

The aim of this study was to evaluate the agronomic usefulness and the biosecurity of the digestate samples derived from the VALORGAS partners (MTT in Finland, Italy, UK and Portugal). The food wastes which the digesters were fed with were also analysed to see the possible differences between the feed characteristics.

## 2 Materials and methods

### 2.1 Origin of materials

Digestates and food wastes used in this study originated from the UK, Italy and Portugal (Table 6). MTT's food waste samples, untreated and autoclaved, were originally from the UK and were sent to MTT Finland, where the FWs were digested in laboratory CSTRs to produce digestates MTT1 and MTT2, as described in VALORGAS deliverables D3.2 and D3.3. MTT1 and MTT2 digestates were combined samples from parallel reactors (R1+R2 and R3+R4) working at an organic loading rate of 4 kg VS m<sup>-3</sup> day<sup>-1</sup>. Samples were collected from the reactor outflow on a daily basis and stored in a refrigerator until the desired amount was obtained (6 litres, stored for a maximum of 2 weeks at 4 °C). After that, samples were stored frozen (-20 °C), and thawed before analysis.

Samples from the UK, Italy and Portugal were sent frozen to MTT, Finland, where they were stored at 4 °C until analysis of the nutrient value, heavy metals and hygienic quality. Before analysing organic composition the samples were frozen (-20 °C) and then thawed again.

**Table 6.** Origin and background information of food wastes and digestates used in the evaluation. Food waste (FW), vegetable waste (VW), waste activated sludge (WAS), organic fraction of municipal solid waste (OFMSW).

Sample	Origin	Scale	Temperature	Phase	HRT (day)	OLR	Feed	
MTT1	UK/MTT	Lab-scale	Mesophilic	1	58	4.0 <sup>a</sup>	FW	
MTT2	UK/MTT	Lab-scale	Mesophilic	1	47	4.0 <sup>a</sup>	Autoclaved FW	
England	Greenfinch	Sub- commercial	Mesophilic	1	25.7	3.3 <sup>a</sup>	Domestic FW	
Italy	Treviso	R1	Pilot	Mesophilic	1	16	3.8 <sup>b</sup>	VW + WAS
		R2	Pilot	Mesophilic	1	24	2.3 <sup>b</sup>	VW + WAS
		R3	Pilot	Thermophilic	1	24	2.3 <sup>b</sup>	VW + WAS
		R4	Pilot	Thermophilic	1	16	3.8 <sup>b</sup>	VW + WAS
		F2	Pilot	Thermophilic	2	13	4.8 <sup>a</sup>	OFMSW
Portugal	Lisbon	Full scale	Thermophilic	2	24	3.7 <sup>b</sup>	OFMSW	

<sup>a</sup> kg VS m<sup>-3</sup> day<sup>-1</sup>

<sup>b</sup> kg COD m<sup>-3</sup> day<sup>-1</sup>

As shown in Table 6 digestate samples were collected from different size digesters with different feedstocks. Samples from Italy were from different anaerobic digesters, samples R1 and R2 from mesophilic reactor and samples R3 and R4 from thermophilic reactors which were all fed with a combination of vegetable waste (VW) and waste activated sludge (WAS). Sample F2 was from 2-phase digestion, the second phase of which was fed with effluent from the first phase (dark fermentation) while the original feed to the process was the organic fraction of municipal solid waste (OFMSW). The OFMSW from Portugal was material selectively collected by Valorsul from the Lisbon area.

Prior to analysis FW samples were macerated with a Retch Grindomix GM300 knife mill (Retch GmbH, Germany). From the Portuguese FW sample also the non-biodegradable material (plastic cups, plastic bags etc.) were removed before analysing the total and soluble nutrients while other analyses (characterisation and hygienic quality) were analysed using the whole sample. The samples and sample amounts used in each experiment are shown in Table 7.

**Table 7.** Samples used in each experiment of the deliverable.

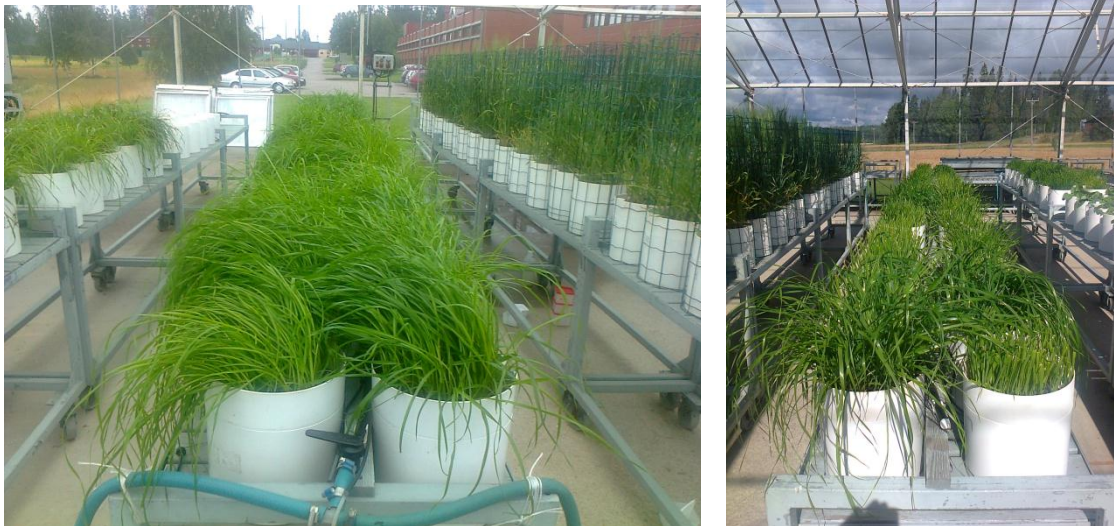
	Characterisation	N mineralisation	Pot experiments	Biosecurity
<b>FWs</b>				
MTT1	x			x
MTT2	x			x
England	x			x
Italy	x			x
Portugal	x			x
<b>Digestates</b>				
MTT1	x	x	x	x
MTT2	x	x	x	x
England	x	x	x	x
Italy R1	x			x
Italy R2	x			x
Italy R3	x			x
Italy R4	x	x	x	x
Italy F2	x			x
Portugal	x	x	x	x

## 2.2 Experimental set-up

### 2.2.1 Pot experiments

Nitrogen availability of digestate samples was tested in pot experiment (Figure 6) with Italian ryegrass (cv. Fabio). The aim was to apply 1500 mg total N in digestates to 5 litres of sandy soil. The application levels were calculated according to the pre-samples and later the actual N analysis of added samples revealed some variation in the applied N levels. Control treatments were inorganic N applications of 0 to 2000 mg N into the pot at 500 mg N intervals. Sufficient levels of P, K and micronutrients were applied to each pot to keep N the only responsive nutrient. Three dry matter yields were harvested at 30, 60 and 160 days after start of the experiment. The fast harvest was after a longer period as the first pots were growing slowly under glass in September, then the growing period was continued in a glasshouse for a further thirty days.

Ryegrass was cut in 2 cm high stubble, fresh weight was measured and the harvested sample dried at 60 °C after which dry weight was determined. Later samples were milled and Kjeldahl N concentrations were determined.



**Figure 6.** Ryegrass growth before the first yield (left) and the second yield (right).

### 2.2.2 Nitrogen mineralisation

N mineralisation tests were run according to ISO 14238 'Determination of nitrogen mineralisation and nitrification in soils and the influence of chemicals on these processes'. The planned addition level of digestates was to apply 20 mg N into 100 g of test soil. According to the available information, 2.2–8.6 g fresh matter was added by different materials. Incubation was done at 20 °C and soil from individual pots was frozen 0, 4, 20 and 48 days after the start of incubation. Soil samples were extracted with 2 M KCl and analysed with a Lachat auto-analyzer for ammonium and nitrate N. Each material was tested with three replicates, and soil inorganic N concentrations were compared against incubated soil without any added fertiliser product.

## 2.3 Analytical methods

### 2.3.1 Hygienic quality

Hygienic quality of the FWs and digestates was analysed using *E. coli*, other coliforms, total coliforms, enterococcus and sulphite-reducing clostridia as indicator organisms. Analyses of coliforms were performed according to Baylis and Patrick (1999) using Harlequin *E.coli*/coliform (LabM) culture medium. Enterococcus were determined with KF streptococcus agar according SFS-EN ISO 7899 (Finnish Standard Association 2000) and sulphite reducing clostridia with sulfite-iron agar according to SFS-EN 26461 (Finnish Standard Association, 1993). For the qualitative analyses of Salmonella samples were pre-enriched in buffered peptone water and incubated in Rappaport-Vassiliadis. Aliquots from the broth were cultured on Salmonella selective Rambach and xylose-lysine-decarboxylase agars. If growth was observed colonies were confirmed with triple sugar iron agar, urea-agar and lysine carboxylase broth (37 °C, 24 h) (ISO, 2002).

### 2.3.2 Chemical analyses

TS and VS were determined according to SFS 3008 (Finnish Standard Association, 1990). TKN was analysed by a standard method (AOAC 1990) using a Foss Kjeltac 2400 Analyzer Unit (Foss Tecator AB, Höganäs, Sweden), with Cu as a catalyst and NH<sub>4</sub>-N according to McCullough (1967). For soluble COD analysis samples food wastes were diluted 1:10 with

distilled water and agitated for 1 hour. After that diluted food waste and raw digestate samples were centrifuged (3500 rpm, 15 min) after which the supernatant was further centrifuged (1320 rpm, 10 min) and stored in a freezer, and then thawed before analysing according to SFS 5504 (Finnish Standard Association 2002a). Total COD was measured by the open reflux, titrimetric method used by the University of Southampton (modified slightly from Vienna standard method). VFAs (acetic, propionic, iso-butyric, n-butyric, iso-valeric, valeric and caproic acids) were determined using a HP 6890 gas chromatograph with an HP 7683 autosampler (Hewlett-Packard, Little Falls, USA) and GC ChemStation Rev. B.03.02 software (column HP-FFAP 10 m x 0.53 x 1.0 mm, carrier gas helium). pH was determined using a VWR pH100 pH-analyser (VWR International).

Crude protein was analysed by Duma's method (AOAC 1990) using a Leco FP 428 nitrogen analyzer (Leco Corp., St. Joseph; MI 49085; USA), where the protein content was determined by multiplying the N% by a factor of 6.25. Soluble carbohydrates were analysed according to Somogyi (1945) and fat content with Accredited In-house methods No. 4.21 and 4.22: Determination by Soxcap-Soxtec-Analyzer (AOAC Official Method 920.39). NDF (Neutral Detergent Fibre) was determined with filtering apparatus according to Van Soest et al. (1991). ADF (Acid Detergent Fibre) and lignin (permanganate-lignin) were determined according to Robertson & Van Soest (1981). Hemicellulose content was calculated from the difference between NDF and ADF, when cellulose content was calculated from the difference between ADF and lignin.

Trace elements were digested in aqua regia according to SFS ISO 11466 (Finnish Standard Association 2007). Approximately 1.0 g of sample was boiled in 9.35 ml of aqua regia for 2 hours, transferred into a 100 ml volumetric flask and filtered. After digestion trace elements (Al, B, Fe, Cu, Cr, Mn, Zn, Ni, Co) were determined with ICP-OES (inductively coupled plasma emission spectrometry, Thermo Jarrel Ash Iris Advantage), and As, Cd, Pb, Mo with GFAAS (graphite furnace atomic absorption spectrometry, Varian AA280Z). Mercury was measured by Mercury Analyzer (Varian M-6000A). The technique is based on cold vapour atomic absorption spectrometry. Mercury was reduced to elemental form with stannous chloride solution and the mercury vapour was lead (using nitrogen as carrier gas) into an absorbance cell for measurement.

Soluble nutrients (N, P, K) were analysed from 1:5 (CEN 13652) and 1:60 water extractions. Samples were shaken for one hour, and then filtered through a cellulose filter (pore size ~ 8 µm). The concentration of NH<sub>4</sub>-N, NO<sub>3</sub>-N and PO<sub>4</sub>-P, were analysed with a Lachat autoanalyzer. Soluble total N in water extractions was measured with a Lachat autoanalyzer after oxidation of organic N into NO<sub>3</sub>-N in autoclave with peroxodisulfate. Soluble total P and K of water extracts were measured with ICP-OES. Total-N was measured by Kjeldahl digestion and total-P with nitric acid digestion as described above.

The measurement of P availability was based on modified Hedley fractionation (Sharpley and Moyer, 2000, Ylivainio et al. 2008), where the fertiliser product was extracted sequentially with water, 0.5 M NaHCO<sub>3</sub>, 0.1 M NaOH and 1 M HCl with a ratio of 1:60. First inorganic P was determined from the extract and then total P concentration was measured after digestion with peroxidase in autoclave. Organic P concentration was calculated as the difference between total and inorganic P.

### 3 Results and discussion

#### 3.1 Food waste and digestate characteristics

Both food wastes and digestates were characterised (Table 8) including the organic composition of materials (Figures 7 and 8). The TS and VS content of the FWs (MTT1, MTT2, England, Italy, Portugal) were quite similar, only the VS/TS relationship in the Italian FW was 15 % lower. The lower organic matter content was also seen in the lower SCOD and COD values of the Italian sample. Total and ammonium nitrogen content was lowest in the Portuguese sample, which can be explained by the composition of the sample: the Portuguese sample was visually detected to include a large amount of plastic material, which has also increased the VS content of that sample. The  $\text{NH}_4\text{-N/TKN}$  ratios were 5-7 % in FWs from MTT, England and Portugal. In the food waste sample from Italy the  $\text{NH}_4\text{-N/TKN}$  ratio was higher (11 %) and the TKN value was lower ( $5.74 \text{ g kg}^{-1} \text{ FM}$ ) compared to other FWs. The SCOD/COD relationship was highest in samples MTT1, MTT2 and England which all originated from the UK. VFA concentrations in all samples were high, possibly partly due to the relatively long storage times and freezing and thawing of materials.

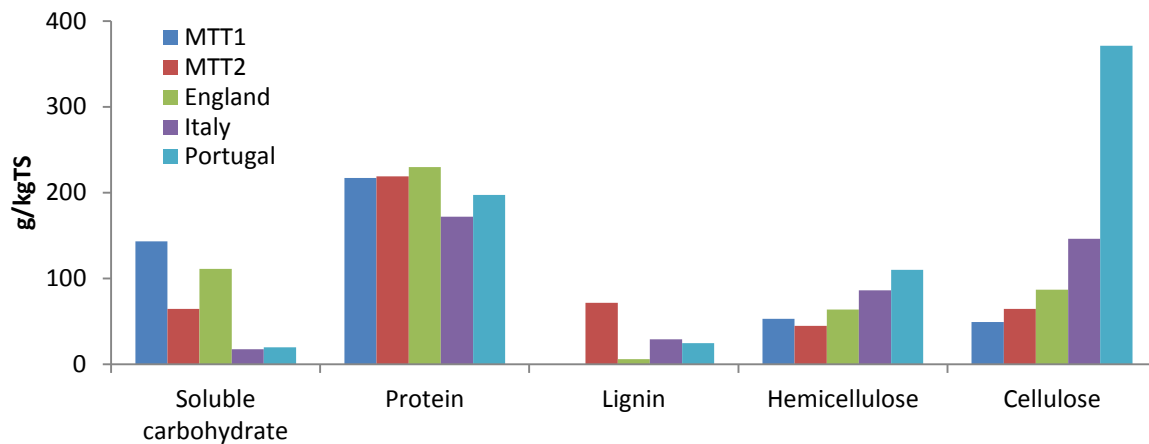
The characterisation of digestate samples showed much more variation compared to FWs. pH values were similar in all samples, but TS and VS contents were notably higher in MTT1 and MTT2 than in the other samples. It might be that in England, Italy and Portugal there was some pre- or after-treatment phase connected to the digestion which diluted the digestate (e.g. wet AD process). MTT1 and MTT2 were digestates from laboratory-scale CSTRs which may also have had some effect on the results. Sample Italy F2 was obtained from the 2-phase reactor where the dark fermentation phase had already used the easily available organic matter, giving a decrease in TS, VS, SCOD and COD. In the Italian samples the effect of HRT can be seen, where the shorter HRT (16 d, R1, R4) shows slightly higher TS and VS while the longer HRT (24 d, R2, R3) has lower TS and VS. The TKN and  $\text{NH}_4\text{-N}$  contents also showed great variation between samples, and the lowest values were obtained in the Italian samples as well as in the MTT2 sample. In the samples from England and Italy F2 almost all nitrogen was in soluble  $\text{NH}_4\text{-N}$  form (82-85 %), which might be related to pre-treatments applied in the process. In other Italian samples (R1-R4) and in the Portuguese sample 60-80 % of nitrogen was in  $\text{NH}_4\text{-N}$  form. In MTT2 the low ammonia content was previously related to the formation of Maillard compounds during autoclaving (see VALORGAS D3.3) which affected the ammonification during digestion. The  $\text{NH}_4\text{-N/TKN}$  ratio in MTT1 was 52 %.

The organic composition of FWs and digestates is presented in Figures 7 and 8. In FWs the protein concentration was alike in all samples while the soluble carbohydrates, fat and fibre concentrations showed some variation. In the Italian and Portuguese samples the soluble carbohydrates and fat concentrations were lower but hemicellulose and cellulose concentrations higher compared to other samples. The variations were likely connected with the FW composition arising from the food consumption and waste separation habits in each country.

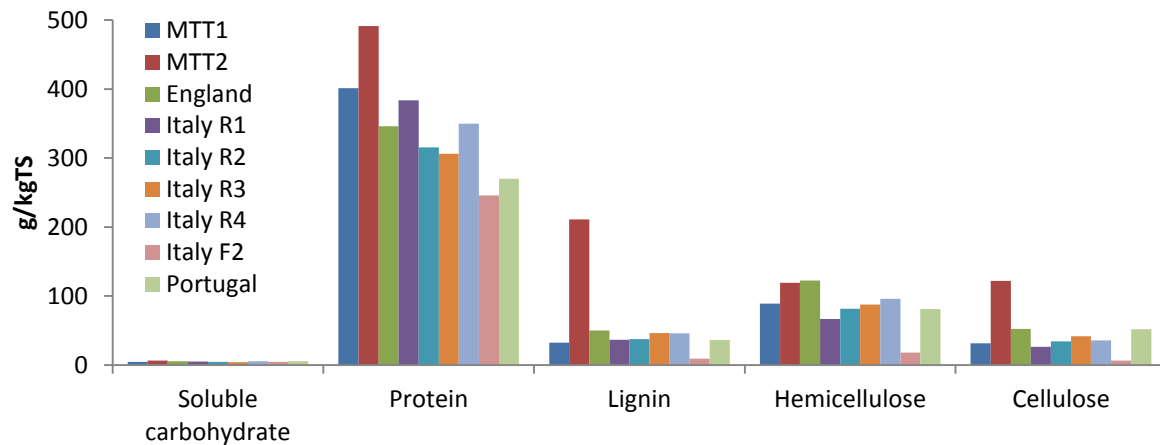
**Table 8.** Food waste and digestate characteristics after freezing and thawing of materials. Results are averages from triplicate analyses from one sample.

	pH	TS (g kg <sup>-1</sup> FM)	VS (g kg <sup>-1</sup> FM)	VS/TS (%)	NH <sub>4</sub> -N (g kg <sup>-1</sup> FM)	TKN (g kg <sup>-1</sup> FM)	C-tot (g kg <sup>-1</sup> FM)	C/N	SCOD (g kg <sup>-1</sup> FM)	COD (g kg <sup>-1</sup> FM)	SCOD/ COD (%)	VFA (g kg <sup>-1</sup> FM)
<i>Food wastes</i>												
MTT1	5.5	247.0	229.9	93.1	0.5	7.8	N/A	N/A	114.6	364.4	31.4	3.1
MTT2	5.4	226.4	209.0	92.3	0.4	7.3	N/A	N/A	104.2	361.2	28.8	2.2
England	5.0	255.1	232.8	91.3	0.6	8.2	N/A	N/A	132.9	444.0	29.9	4.9
Italy	5.4	271.1	208.6	76.9	0.7	6.4	N/A	N/A	78.4	339.8	23.1	4.8
Portugal	4.7	287.0	264.3	92.1	0.3	5.7	N/A	N/A	69.9	412.5	17.0	5.5
<i>Digestates</i>												
MTT1	8.0	68.1	50.2	73.6	4.5	8.7	26.9	3.1	15.4	77.1	20.0	3.3
MTT2	7.6	78.8	63.7	80.9	1.7	7.8	25.9	3.3	18.5	100.3	18.4	1.1
England	8.3	19.9	12.3	61.7	3.9	4.7	6.8	1.5	11.2	21.8	51.4	4.1
Italy R1	6.7	33.7	24.6	73.0	1.1	1.9	12.3	6.6	6.9	25.0	27.8	5.0
Italy R2	7.7	23.3	16.4	70.3	1.0	1.3	8.0	6.3	2.5	12.5	19.7	1.1
Italy R3	8.1	18.6	12.6	67.5	1.3	1.8	6.4	3.6	3.1	7.6	41.1	1.1
Italy R4	7.6	34.2	23.9	69.9	1.7	2.2	13.5	6.1	8.4	26.7	31.5	3.4
Italy F2	8.4	7.2	4.1	57.2	1.2	1.4	1.8	1.3	3.2	5.8	55.6	0.3
Portugal	8.3	32.2	18.9	58.7	3.2	4.5	10.3	2.3	7.3	30.6	23.9	0.3





**Figure 7.** Organic composition of food wastes. Results are averages from triplicate analyses from one sample.



**Figure 8.** Organic composition of digestates. Results are averages from triplicate analyses from one sample.

When the organic composition of FWs was compared with the digestates, it was observed that soluble carbohydrate and fat concentration in all digestates was decreased due to digestion. Protein concentration increased as a result of the microbial biomass produced in the digestion process, while the fibre concentrations showed some variation. When the Italian samples were compared the hemicellulose, cellulose and lignin were observed to be higher in thermophilic digestates (Italy R3, R4) compared to the mesophilic (Italy R1, R2). The Portuguese sample also originated from a thermophilic reactor, but the fibre concentrations were very close to those from the mesophilic samples MTT1, MTT2 and England. The cellulose content in the Portuguese FW was very high, over 4 times higher than in other samples, but the same difference was not seen in the digestate sample. The Italian sample F2 showed remarkably lower values in the case of fibre which is likely related to the 2-phase reactor performance.

The water soluble nutrient content was determined with 1:5 and 1:60 water extractions from the digestate samples (Table 9). Overall 1:60 water extraction resulted in higher values but with a few samples water soluble total nitrogen and  $\text{NH}_4\text{-N}$  in the 1:5 extraction resulted

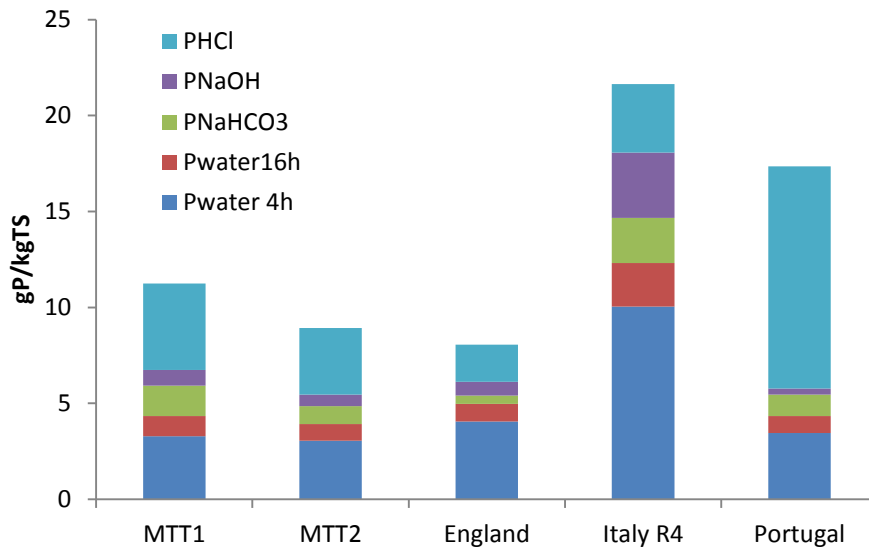
higher values. In samples MTT2 and Italy R1, R3, R4 and F2 both  $\text{NH}_4\text{-N}$  and tot-N resulted in slightly higher values with 1:5 extraction while in samples England and Italy R2 tot-N was slightly higher with 1:5 extraction than with 1:60 extraction. Highest total-N values were obtained from samples MTT1, England and Portugal where the water soluble nitrogen concentrations were over  $4 \text{ g kg}^{-1}$  FM; while in Italian samples the soluble tot-N remained under  $2 \text{ g kg}^{-1}$  FM due to the low total solids in the samples. The autoclaved MTT2 sample however, gave  $3 \text{ g kg}^{-1}$  FM of soluble total-N which was under 40 % of the total Kjeldahl nitrogen from the whole sample (in Table 8). This indicates that during the anaerobic digestion of the autoclaved FW, the nitrogen containing molecules, proteins, cannot be solubilised which eventually affects the conversion efficiency of the process leading to reduces methane yields as was observed in VALORGAS D3.3.

**Table 9.** 1:5 and 1:60 water soluble nutrients in digestates. All values are concentrations in water soluble form.

	MTT1	MTT2	England	Italy R1	Italy R2	Italy R3	Italy R4	Italy F2	Portugal
<b>1:5 water soluble (<math>\text{g kg}^{-1}</math> FM)</b>									
soluble tot-N	5.99	3.01	4.44	1.83	1.32	1.86	2.23	1.57	4.02
$\text{NH}_4\text{-N}$	4.35	1.94	3.32	1.14	1.00	1.39	1.59	1.22	2.77
$\text{NO}_3\text{-N}$	0.013	0.011	0.011	0.000	0.002	0.002	0.003	0.003	0.007
$\text{PO}_4\text{-P}$	0.27	0.14	0.06	0.23	0.19	0.23	0.35	0.01	0.13
tot-P	0.33	0.19	0.11	0.30	0.20	0.23	0.35	0.02	0.15
tot-K	3.24	2.50	1.87	0.47	0.31	0.44	0.58	0.75	1.89
<b>1:60 water soluble (<math>\text{g kg}^{-1}</math> FM)</b>									
soluble tot-N	6.15	2.91	4.20	1.25	1.01	1.37	1.88	1.27	4.40
$\text{NH}_4\text{-N}$	4.89	1.71	3.78	0.97	0.90	1.20	1.46	1.11	3.14
$\text{NO}_3\text{-N}$	0.014	0.013	0.012	0.002	0.003	0.004	0.002	0.004	0.008
$\text{PO}_4\text{-P}$	0.39	0.17	0.10	0.28	0.23	0.27	0.43	0.01	0.17
tot-P	0.47	0.22	0.12	0.35	0.25	0.31	0.45	0.03	0.21
tot-K	3.56	2.54	1.97	0.47	0.32	0.45	0.60	0.77	2.11

The total water soluble phosphorous (tot-P) and phosphate ( $\text{PO}_4\text{-P}$ ) analysed were consistently higher with 1:60 extraction than with 1:5 extraction. In sample Italy R4 95 % of the water soluble total-P consisted of  $\text{PO}_4\text{-P}$  while in samples R3 and R3 the  $\text{PO}_4\text{-P}/\text{tot-P}$  ratio was 89 and 88 %. The phosphate to tot-P ratio was around 80 % with samples MTT1, MTT2, England, Italy R1 and Portugal while in Italy F2 the ratio was only 50 % likely due to the 2-phase process configuration. However, the water soluble potassium showed very similar results at both extraction ratios.

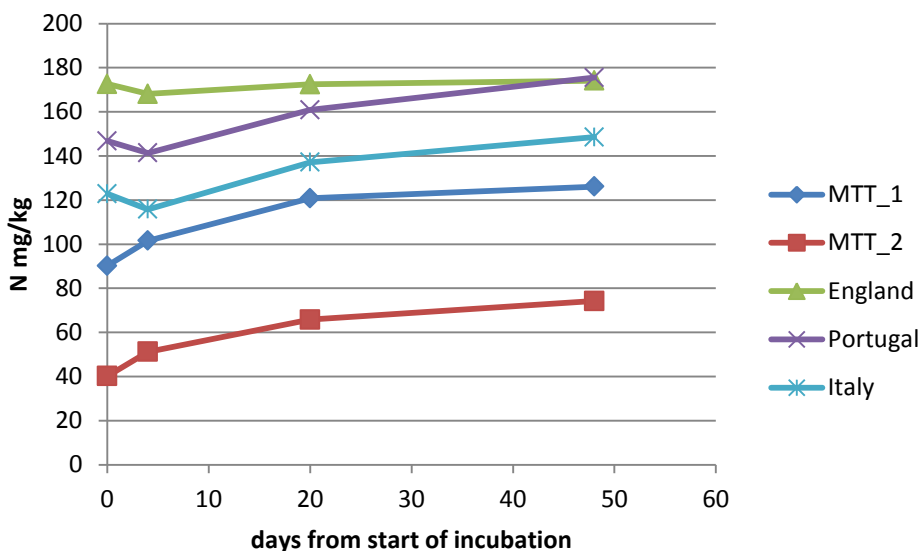
The plant available P was analysed with Hedleys fractionation (Figure 9). A considerable proportion of P was water soluble, except in the Portuguese digestate. The sum of water and  $\text{NaHCO}_3$  extractable P (Olsen-P) can be considered as plant available and this was 50-70 % in samples of MTT, Italy and England. When phosphorus is from organic sources it can be expected to be readily soluble and thus readily available for plants. The lower P solubility of sample from Portugal suggests that the input is different from the other tested FW digestates.



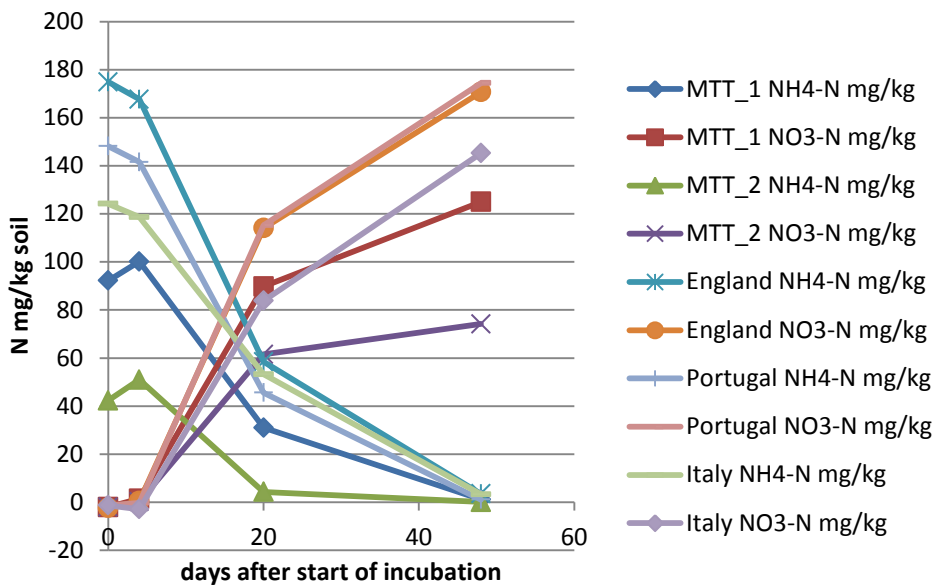
**Figure 9.** Phosphorus solubility determined with Hedleys fractionation.

### 3.2 N mineralisation and pot experiments

In the N mineralisation experiment the planned application rate of 200 mg total N kg<sup>-1</sup> of soil varied, as the applied materials had slightly different N concentrations as expected (Table 10). All materials contained considerable amount of NH<sub>4</sub>-N. Application of dissolved organic N (DON) of 1:5 water extractions was 30–60 mg kg<sup>-1</sup> soil and this proportion of organic N can be considered most easily mineralised. Mineralisation of organic N was of the same magnitude (30 N mg kg<sup>-1</sup> soil) from all other digestates except the digestate from England (Table 11 and Figure 10). Nitrification of NH<sub>4</sub>-N to NO<sub>3</sub>-N happened at a fast rate after all digestate applications (Figure 11). Considering the fertiliser value, the digestate from England responds to its NH<sub>4</sub>-N concentration. Other digestates had lower ammonium concentrations and 15–30% of their organic N mineralised during the incubation.



**Figure 10.** Nitrogen mineralisation of digestates in the incubation experiment.



**Figure 11.** Soil ammonium (NH<sub>4</sub>-N) and nitrate (NO<sub>3</sub>-N) concentrations during the incubation.

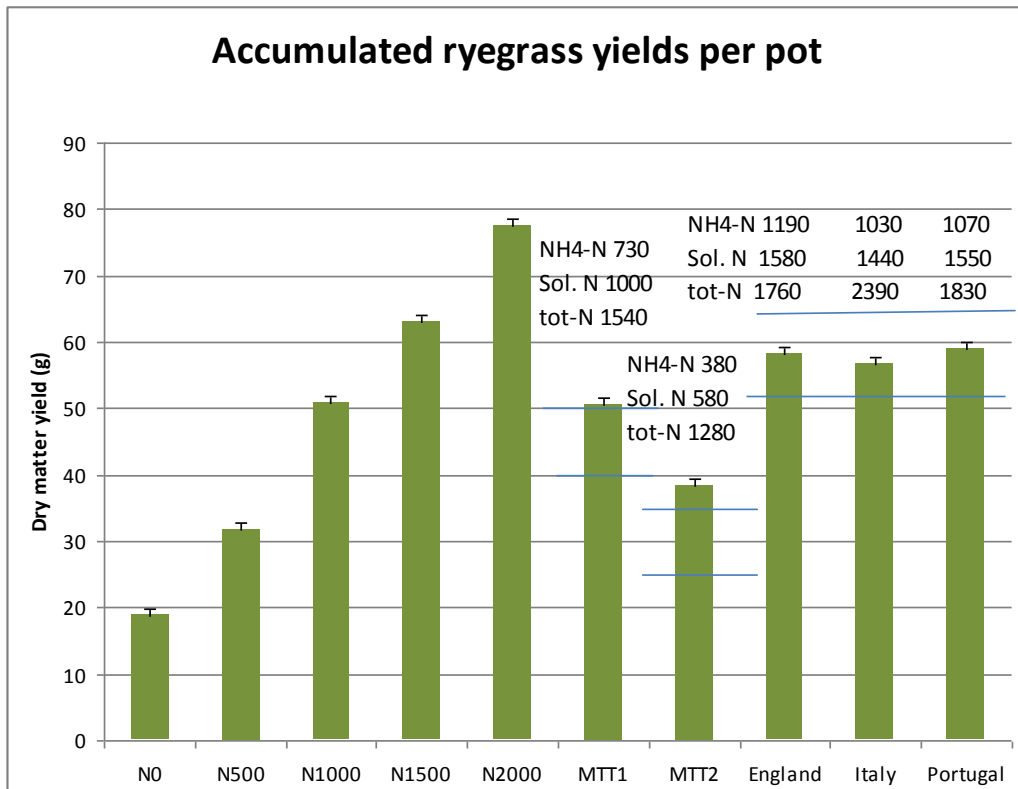
**Table 10.** Applied fresh matter, nitrogen components and mineralisation of organic N in the incubation test.

	Application			Applied mg kg <sup>-1</sup>			Mineralisation from organic N		
	FM g per 100g	Total-N	Organic-N	DON	NH <sub>4</sub> -N	NO <sub>3</sub> -N	mg kg <sup>-1</sup>	% of DON	% of organic-N
MTT1	2.2	205	108	36	97	0	36	100	33
MTT2	2.6	171	121	27	50	0	34	125	28
England	4.8	235	77	53	158	1	2	3	2
Portugal	5.1	244	102	64	142	0	29	45	28
Italy R4	8.6	318	181	54	137	0	26	47	14

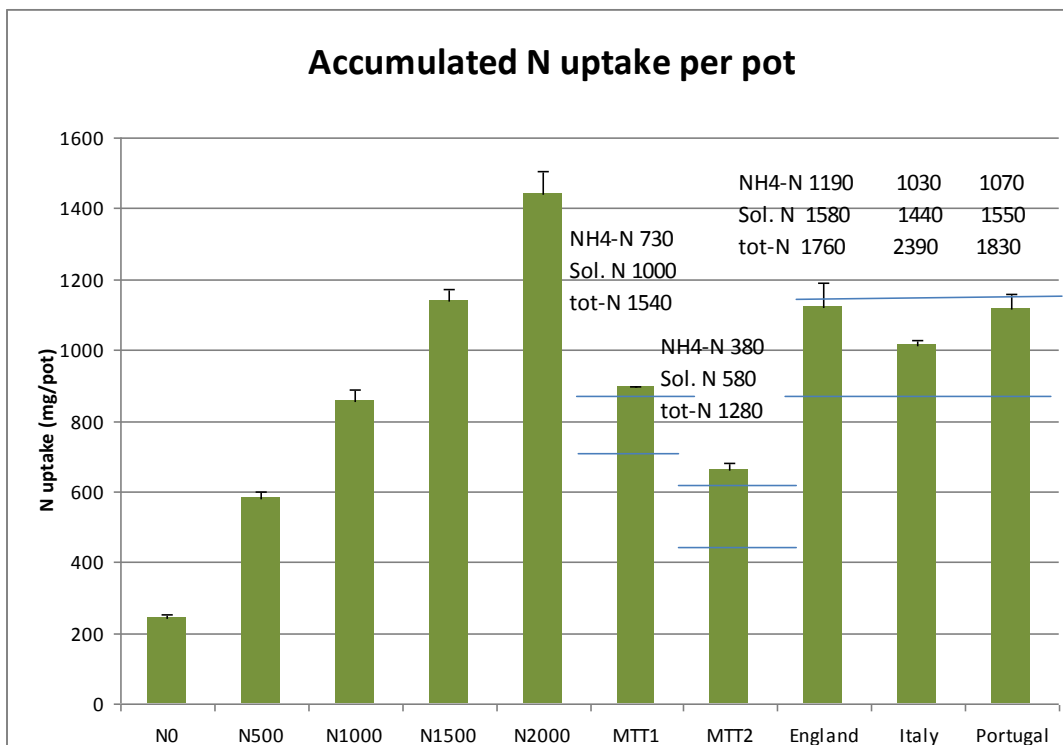
DON = organic N dissolved in 1:5 water extraction

The N uptake in pot experiments was executed with Italian ryegrass (cv. Fabio) (Figures 12 and 13). Applications of digestate NH<sub>4</sub>-N, soluble N and total N are shown above each digestate bar. Blue vertical lines show the respective yield level of inorganic fertiliser application compared to NH<sub>4</sub>-N (lower) and soluble N (higher line). Error bars show standard deviation of the treatments.

All digestate applications produced better yield than could be estimated according to their NH<sub>4</sub>-N content. Digestates from England, Italy and Portugal increased ryegrass yield only slightly more than expected according to the NH<sub>4</sub>-N content. Soluble N was not fully available from those digestates. Digestates MTT1 and MTT2 seemed to release all of their soluble N as yields were comparable to their soluble N content.



**Figure 12.** The effect of digestate N on ryegrass dry matter yield in the pot experiment.



**Figure 13.** Ryegrass N uptake in the two first yields of the pot experiment.

### 3.3 Biosecurity

#### 3.3.1 Heavy metals and trace elements

The metal concentrations in food wastes and digestates were studied from dried samples. In food wastes, concentrations between samples varied greatly thus e.g. cadmium and mercury concentrations were low ( $<0.1 \text{ mg kg}^{-1}$ ) in all samples (Table 11). In the autoclaved sample MTT2 higher concentrations of Pb, Hg, Al, Mo, B, Fe, Cu, Zn, Mn, Cr and Co were observed compared to sample MTT1, possibly due to residues from the autoclaving apparatus or to the release of metals during pre-treatment.

**Table 11.** Metal concentrations in food wastes.

mg kg <sup>-1</sup> DM	MTT1	MTT2	England	Italy	Portugal
Pb	0.20	2.21	0.65	3.35	0.49
Ni	0.64	0.47	1.03	4.21	0.76
Hg	0.06	0.08	0.08	0.09	0.05
Cd	0.06	0.05	0.06	0.10	0.02
Al	100	120	160	3590	140
Mo	0.50	0.63	0.44	1.32	0.20
B	10.1	22.0	8.5	24.4	13.0
As	0.45	0.45	0.39	1.41	0.22
Fe	0.13	7.12	0.35	5.61	0.28
Cu	4.93	8.35	5.74	16.56	9.61
Zn	28.2	37.8	29.4	41.5	93.3
Mn	78.1	154.8	69.9	126.0	8.8
Cr	1.09	3.25	1.76	18.74	1.31
Co	0.14	0.37	0.21	0.85	0.40

From food wastes to digestates the metal concentrations increased which indicated solubilisation of metals during digestion, and also the reduction in solids content during digestion. Concentrations of mercury, copper, lead and aluminum were notably higher in the Italian samples R1-R4 (Table 12) compared to other samples. This might be due to the different origin of these samples (feed material: VW+WAS). The cadmium concentration was highest in Italian samples R1-R4 but also in the Portuguese sample ( $\sim 1 \text{ mg kg}^{-1}$ ) while the highest cobalt concentrations ( $10\text{-}30 \text{ mg kg}^{-1}$ ) were observed with samples UK, MTT1 and MTT2, all originating from the UK.

The usability of the digestates as fertiliser in agriculture was studied by comparing the metal (Pb, Ni, Hg, Cd, Cu, Zn, Cr) concentrations with the limit values from national legislations. For trace elements (Al, Mo, B, Se, Fe, Mn, Co) there were no limit values. For arsenic limit values were implemented only in Finnish legislation. The Portuguese legislation proposal for compost had the lowest limit values for heavy metals (class I) while in Finland the limiting values allowed the highest heavy metal concentrations in digestates (Table 3).

**Table 12.** Metal concentrations in digestates.

mg kg <sup>-1</sup> DM	MTT1	MTT2	England	Italy R1	Italy R2	Italy R3	Italy R4	Italy F2	Portugal
Pb	2.10	5.60	5.63	79.67	98.67	99.21	98.04	6.64	11.66
Ni	17.78	16.64	42.39	19.69	24.76	26.50	22.25	10.42	6.69
Hg	0.12	0.19	0.09	1.77	2.49	2.41	1.81	0.16	0.29
Cd	0.23	0.13	0.26	0.88	1.39	1.28	1.11	0.32	1.50
Al	870	440	560	6350	8940	8410	7680	2040	2050
Mo	4.77	3.80	8.39	6.65	8.53	8.58	7.61	3.82	3.30
B	33.7	27.2	34.8	70.0	61.5	68.1	65.7	65.4	55.6
As	0.70	0.44	1.01	1.90	2.99	2.81	2.59	3.09	3.32
Fe	4.52	6.16	2.32	5.42	6.60	6.41	5.91	5.25	3.74
Cu	25.64	22.37	21.69	551.60	512.10	522.60	626.50	34.83	58.70
Zn	116.0	94.6	175.0	800.0	1099.0	1056.0	1006.0	140.0	401.0
Mn	283.7	230.2	77.6	95.9	110.5	109.3	105.3	139.1	105.4
Cr	9.82	11.89	7.48	29.09	37.29	35.72	32.89	23.51	12.97
Co	13.09	11.24	31.28	1.64	2.35	2.51	2.19	0.97	1.45

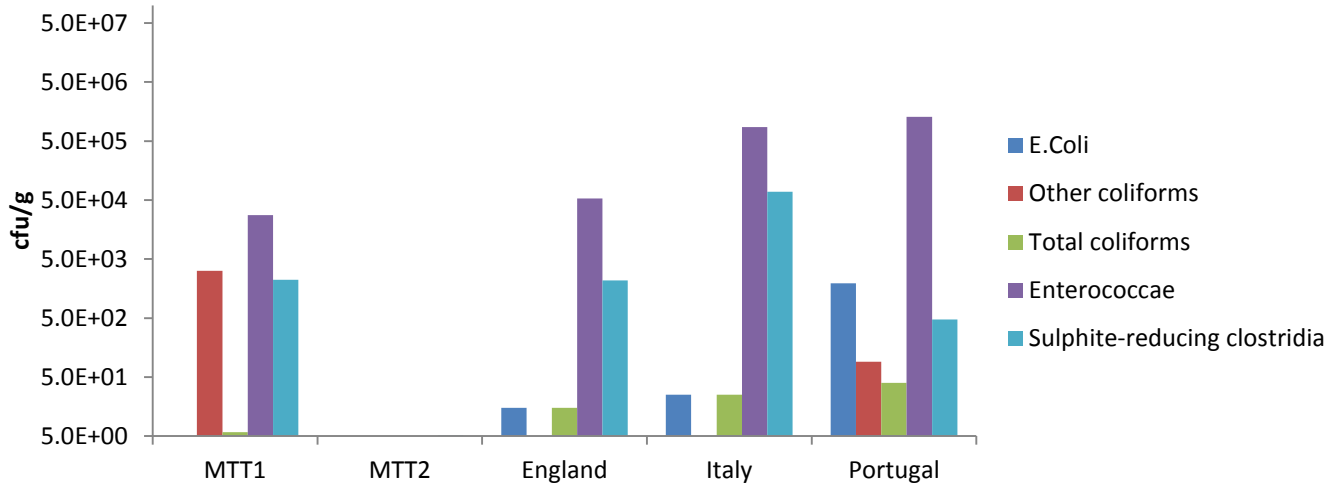
All the studied digestates were below the limiting values of lead, nickel and chromium. Samples MTT1, MTT2, UK and Italy F2 had all metal concentrations under the limiting values appropriate for fertiliser materials in Portugal (if composted after digestion), Finland and UK. Because Portuguese legislation proposal for compost (class I and II) and digestate legislation in UK have low limiting values for mercury, cadmium, copper and zinc, samples Italy R1-R4 were not suitable as fertiliser materials according to these limits. The zinc concentration in the Portuguese sample exceeded the Portuguese class I limit (200 mg kg<sup>-1</sup>) and was very close to the limit value in UK (400 mg kg<sup>-1</sup>). A limit for arsenic was implemented only in Finnish legislation for organic fertilizers, but none of the samples exceeded the limit (25 mg kg<sup>-1</sup>). It should be noted that the very high degradability of food waste can give relatively high concentrations of metals in digestate when quoted on a dry matter or organoc dry matter basis, rather than a wet weight basis, due to the high degree of solids degradation achieved compared to less degradable wastes.

### 3.3.2 Hygienic quality

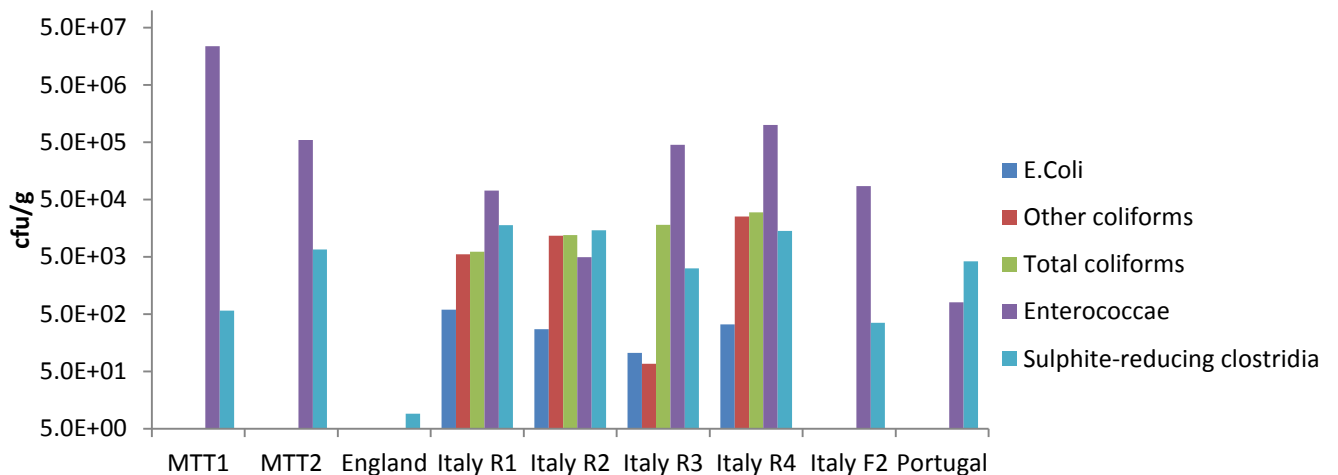
The hygienic quality of the food wastes and digestates was determined with hygiene indicators *E. coli*, other coliforms, total coliforms, Enterococcae, Sulphate reducing clostridia and Salmonella (Figures 14 and 15). Because samples were shipped frozen and were stored few days in the freezer before analysis, the results cannot be related to results from fresh samples. However, no Salmonella was detected in neither FW nor digestate samples. Enterococcae was found in similar concentrations in all FW samples except the autoclaved MTT2 sample, where the autoclave treatment had sterilised the material very efficiently. Coliforms were detected in all samples except MTT2.

As in the FW samples also in the digestate samples the concentration of Enterococcae remained quite high (except the UK sample), and in both MTT samples the concentration was notably increased compared to the FWs. This phenomenon is likely due to the microbial population from the inoculum of MTT's reactors. In UK there is some hygienisation process prior or after the digestion process because almost no hygiene indicators were found in the

sample from England. Italian samples R1-R4 showed diverse hygiene indicator population which slightly reflected the temperature and retention time applied to each reactor. Other coliforms were detected in the Italian digestates (R1-R4) but not in the Italian FW: this was due to the different origin of R1-R4 feed material (WAS+VW) which was not analysed in this experiment. The Italian F2 sample obtained from the second phase of the 2-phase process contained only Enterococcae and Clostridia as well as the Portuguese sample, likely due to the thermophilic conditions applied during the anaerobic digestion.



**Figure 14.** Hygienic quality of food wastes (after freezing and thawing).



**Figure 15.** Hygienic quality of digestates (after freezing and thawing).

According to EU regulations concerning animal by-products (EC 1069/2009 and EU 142/2011) and their digestion residues the standards for the threshold values for *E. coli* or Enterococcaceae are 1000 cfu g<sup>-1</sup> when no Salmonella is detected in 25 g sample. According to this legislation the samples MTT1, MTT2, England, Italy F2 and Portugal could be used in agriculture because no Salmonella was detected and the concentration of *E. coli* remained under the threshold value. However, Italian samples R1-R4 would not be appropriate for agricultural use without a hygienisation step.



## 4 Conclusions

Food waste treated in anaerobic digesters to produce organic fertiliser for agricultural use enhances the nutrient cycle in the food production and food waste chain. Food waste collected from municipal sources may, however, contain impurities which can affect the end-use of the digestate. The food waste may be contaminated by organic, inorganic or physical impurities which can affect the fertiliser value, pose human and animal health risks, or complicate the digestate spreading. FW may also be contaminated with pathogens which according to EU legislation must be destroyed before or after the anaerobic digestion process to provide a safe end product.

Use of FW as an organic fertiliser in agriculture is highly controlled by legislation in Europe. The legislation is based on the EU's framework directives which are implemented by each country in their national legislation. There are, however, major differences between countries, e.g. Finland, UK, Italy and Portugal which were studied in this report. Application of digestate is allowed in Finland and in the UK, while in Italy and Portugal only composted digestate is suitable for fertiliser use. Also the implementation of EU guidelines varies, e.g. in Finland the soluble-N limit per hectare per year (170 kg) is applied to all digestates irrespective of the origin of the feed material. In UK the limit is applied only to manure-based digestates, as stated in the EU directive (EEC 676/01). Also the application of phosphorus to land is regulated differently in each country and the regulations in Finland are dependent on the feed material of the anaerobic digester. National legislation is essential, however, when it comes to national and regional climate, soil and crop types, which vary between countries. The soil and climate conditions affect the applicable N fertiliser capacity and are thus regulated by each country.

Under the UK waste regulations the EA requires digestate spreading permissions to be in place well before spreading commences. This can be both wasteful and expensive but it is vital that permission is in place to make use of all spreading opportunities. This bottleneck is removed by product status. Digestate has the potential to replace mineral fertiliser nutrients for nitrogen, phosphate and potash. However the presence of significant quantities of crop-available nitrogen especially means that digestate has to be used carefully both to give maximum agricultural benefit and to avoid causing damage to the environment. Processing of digestate in order to concentrate the nutrients or improve the characteristics of the digestate would clearly offer major benefits in managing digestate, but the processes would need to be both economic and reliable. These processing techniques include solid-liquid separation processes with centrifuges or belt presses. The produced solid fraction can be further pelletised or used as such, if that is allowed in the current legislation. The solid fraction can also be composted to produce soil amendment material, if the legislations require this treatment. The liquid fraction could be used as such, or further processed to produce fertiliser products with e.g. ammonia stripping or struvite precipitation (see also VALORGAS deliverables D3.6 and D4.7).

If digestate is used to achieve best agricultural benefit the spreading of digestate is necessarily highly seasonal. This poses major logistical pressures for AD operators, which can be mitigated by the use of on-farm storage and spreading facilities. Storage of the digestate is essential when it comes to limited fertilising seasons, and can be executed with different technologies e.g. floating covers on top of the storage tanks to prevent gaseous emissions. Some of the logistical problems could be prevented by using e.g. solid-liquid

separation, reducing the amount of transported water and concentrating the nutrients which also reduces the price for the transport. Application of digestate must be well planned to prevent environmental problems caused by the volatilisation of  $\text{NH}_3$  during spreading and to provide plants with the effective amount of nutrients.

The agricultural usefulness and the biosecurity aspects of digestates from different AD plants involved in the VALORGAS project were analysed. The results showed that the digestates studied had potential as fertiliser as such, without any pre-treatment step such as solid-liquid separation. The characteristics of digestates varied in terms of TS concentration due to variation in the AD reactor configurations. Thus it would be efficient to pre-treat the digestate samples from Italy to decrease the water content of the digestates and the applicable amount per hectare. Italian samples (R1-R4, originating from vegetable waste and WAS) also had higher heavy metal concentrations making these samples not appropriate for agricultural use according to the stricter Portuguese (for composted digestate) and UK legislation. However, digestates from food waste were all suitable fertiliser materials. Also in the hygienic quality analysis Italian digestate samples were the only ones not to pass the EU regulations on *E. coli* and enterococcus concentrations; this, however, could be overcome by a hygienisation step before or after digestion. All in all, the food waste digestates had good biosecurity in the case of heavy metals and pathogens.

When the nutrient availability to plants was tested the sample MTT2, derived from FW generated in UK and pre-treated by autoclaving, showed reduced nitrogen availability which was connected with the effects of the autoclave treatment on the waste. All digestates studied showed potential to be used in agriculture with just few adjustments regarding the biosecurity and transportability of materials.

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