Annex 6b:

Agronomic Trial Protocol to Assess Fertiliser Value of the Recycled Derived Fertilisers

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Abstract

Increasing the use of organic and waste-based recycling-fertiliser products can increase the recycling rates of valuable plant nutrients (N/P/K) and C sequestration for sustainable agricultural intensification, which facilitate closing the nutrient cycle loop within the agro-industrial food supply chain. In this context, a number of recycling-derived fertiliser (RDF) candidates like dairy food processing sludge from dairy industry, struvite (phosphate mineral) from sewage and potato wastewater processing and P rich ash from sewage sludge and poultry litter, and cattle slurry (commonly used organic fertiliser) are currently being investigated for their P mineral fertiliser equivalent (P MFE), balance fertiliser efficiency and agronomic quality under European Commission funded Interreg project "ReNu2Farm" - increasing the recycling rates of N, P and K. The trial was set out in grassland agronomic condition with low P index grassland soil under two experimental set up – 1) P MFE plots and 2) balance fertilizer application plots. The results from this investigation will demonstrate the performance of the mentioned RDFs on nutrient supply for crop production in comparison to their counter-trial using mineral-based synthetic fertilisers. The outcome would inform the different stakeholders (farmers, recycling-based SMEs) about the potential scope of increasing nutrient recovery and recycling from different agri-food processing based waste resources and thus facilitates to replace mineral fertilisers.

Keywords: Nutrient recovery and recycling; Renewable fertilizer; Grassland; Agronomy

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1. Scope and context

The purpose of this field trial was to determine if recycled derived phosphorous (P) fertilizers could replace mineral P fertilizers in an effort to increase the sustainability of farm systems, close off P loops and reduce the amount of farm income spent on fertilizer. There is growing demand to recover and recycle P from secondary resources like waste stream in order to achieve sustainable global food security (Huang et al., 2017), a priority for the future development agenda 2030 of the United Nations (UNDP, 2016). In this context, P recovery technologies are particularly focused on utilising alternative sources such as manure, sewage, or wastewater. For example, struvite (magnesium ammonium phosphate, $MgNH_4PO_4 \times 6H_2O$), a P recovery product from wastewater, has gained interest as a low soluble slow release P fertiliser with full-scale operation of struvite production (e.g. Ostara struvite-crystallisation processes Pearl, AirPrex and NuReSys) available in Europe (Hukari et al., 2016). The technical recovery of P from municipal wastewaters is approximately 2000 tonnes/year in Europe (Hukari et al., 2016). In addition, P from partially to fully dried sludges and/or manure can be recovered by chemical leaching, (hydro)thermal treatment, composting, and mono-incineration for application as fertilizer (Bradford-Hartke et al., 2015). In the recent years, a number of studies looked into the transformation of P in the thermally treated solid biowastes/sewage sludge and animal manures to assess its availability for fertilisation (Huang et al., 2017, 2018), which suggests that P speciation and availability are highly dependent on treatment techniques and conditions that are adjustable to enhance P recovery and recycling from different biowastes. It means agronomic demonstration trial is very important to investigate the fertiliser efficiency and agronomic quality of recycling derived P fertilisers in comparison to the mineral-based synthetic P fertilisers (e.g. triple supper phosphate).

In the present study, field-scale agronomic trial was designed to assess the P fertiliser value and crop yield performance of a number of recycling-derived fertiliser (RDF) candidates under two experimental set up – 1) P Mineral Fertilizer Equivalent (P MFE) plots and 2) balance fertilizer application plots. The RDF products were (Figure 1) – 1) struvites (one from potato processing wastewater) and another from sewage sludge), 2) ash (one from poultry litter and another from sewage sludge), 3) dairy food processing sludge, and 4) cattle slurry. The P MFE trial will identify the plant available fraction of the P in the listed RDF products with respect to their total P content and balance fertiliser application trial will generate better knowledge on the economic and agronomic performance of RDF products comparing synthetic mineral fertilisers (Figure 2 shows triple super phosphate containing 16%).

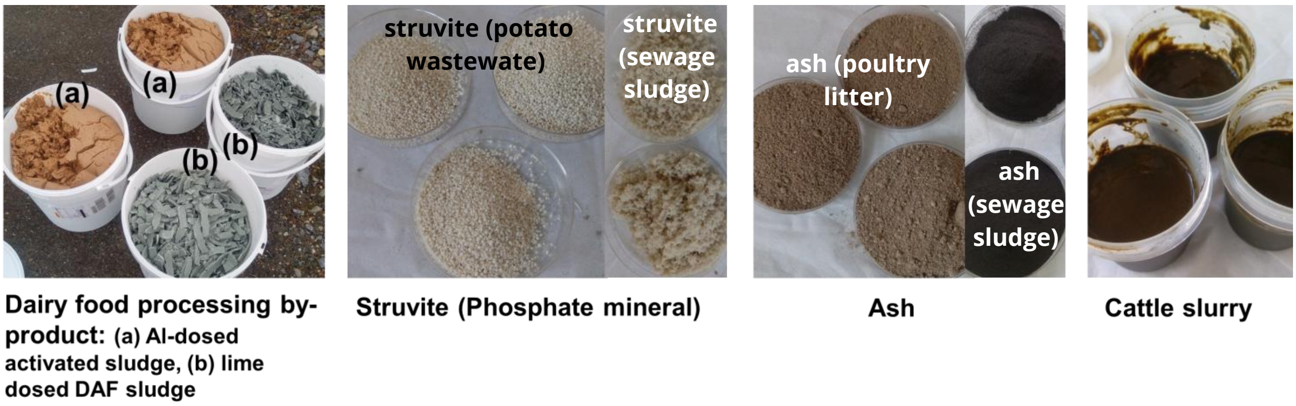

Figure 1 Recycling derived fertiliser products for agronomic trial.

Figure 2 Mineral fertilizer triple super phosphate (16% P).

2. Set-up of demonstration trial

2.1. Plot plan, trial design and application rates

The trial was set up in a site which had been reseeded with a crop of perennial rye grass and which soil tests had shown to have residual phosphorous (P) concentrations in the soil up to 3 mg/L (i.e. P index 1 soil site). This was ideal as it meant there was scope to apply a high rate of P with likely chance that the available P would be taken up by the grass. In general, P is considered to be the limiting factor to estimate a legal application rate (usually 40 kg-P/ha for pasture establishment at a low Morgan's P Index soil (e.g. P Index 2 equivalent to Morgan's P of 3.1–5.0 mg/L, where Morgan's P indicates plant available P in soil)) when applying biosolids and cattle slurry as organic fertiliser (Ashekuzzaman et al., 2018). The lime requirement (determined from pH analysis of soil) was also high, which indicated a pre-trial application of 1.5 ton/ha of lime.

The plot layout and plan is shown in Figure 3 and Figure 4. The trial plots were divided into two sets of experiments – 1) P mineral fertilizer equivalent (MFE) (treatment number 1−14 in Figure 3) and 2) balance fertilizer application (treatment number 15−23 in Figure 3). The treatments for the P MFE experiment were – zero fertilizer (control plot without any fertilizer application), plot with different application rate of P fertilizer (0 – 60 kg/ha), plot with different RDFs (with P application rate of 40 kg/ha) that included dairy processing sludge (DAF and activated), cattle slurry, struvite (sewage and potato processing wastewater) and ash (sewage sludge and poultry manure) (treatments are shown in Table 1). There was application of N, K and S fertilizer in all P MFE plots @ 125, 155 and 20 kg/ha, respectively. For the balance fertilizer trial, all RDFs were also applied @ 40 kg P/ha and any requirement of additional supply of N, K and S were balanced by mineral fertilizer application. The application rates can be seen in Table 1. For chemical fertilizer, the source of N, P, K and S was calcium ammonium nitrate (CAN), triple super phosphate, muriate of potash (MOP) and sulfur of potassium (SOP), respectively. The application rates for N, P, K and S were followed as per recommendation from Tegasc nutrient advise reference book (Teagasc Greenbook, 2016). Based on such application rates all mineral and RDFs were weighed out using calibrated balance as shown in Figure 5.

Plot 6m X 2m
2m bufferzone between plots

Entrance

8 m | 38 m

P-MFE trial

Field Road | 53.5 m | 46 m = 23@ 2m

		6	2	6	2	6	2	6	2	6
1	101	10	201	12	301	8	401	3	501	4
2	102	14	202	10	302	4	402	8	502	13
3	103	9	203	2	303	1	403	9	503	6
4	104	1	204	13	304	11	404	7	504	9
5	105	2	205	7	305	5	405	12	505	5
6	106	3	206	5	306	13	406	11	506	12
7	107	4	207	14	307	9	407	14	507	3
8	108	5	208	11	308	7	408	2	508	10
9	109	6	209	9	309	14	409	10	509	11
10	110	7	210	8	310	6	410	13	510	8
11	111	12	211	4	311	10	411	1	511	14
12	112	8	212	6	312	2	412	5	512	1
13	113	11	213	3	313	12	413	4	513	7
14	114	13	214	1	314	3	414	6	514	2
15	115	16	215	23	315	15	415	23	515	15
16	116	21	216	17	316	18	416	16	516	19
17	117	17	217	20	317	22	417	19	517	22
18	118	19	218	21	318	16	418	22	518	23
19	119	15	219	22	319	20	419	17	519	18
20	120	23	220	18	320	21	420	20	520	16
21	121	22	221	16	321	17	421	18	521	21
22	122	20	222	15	322	19	422	15	522	20
23	123	18	223	19	323	23	423	21	523	17

Figure 3: Plot plan operation showing plots 101-523 with treatments 1-23. Plots measure 6×2 m^2. 5 Replicates are separated by 2 meter alleys.

Figure 4 Grassland plots (115 plots @ 6×2 m2) with randomly allocated fertiliser products.

Table 1 Chemical and RDFs fertiliser treatments and their application rates.

		Mineral fert application (kg/ha)				Biofert application (kg/ha)				Total Applied (kg/ha)			
Treatments	**Fertilisers**	N	P	K	S	**N**	**P**	**K**	**S**	N	**P**	K	S
1	Zero fertiliser	0	0	0	0	0	0	0	0	0	**0**	0	0
2	P0	125	0	155	20	0	0	0	0	125	**0**	155	20
3	P15	125	15	155	20	0	0	0	0	125	**15**	155	20
4	P30	125	30	155	20	0	0	0	0	125	**30**	155	20
5	P40	125	40	155	20	0	0	0	0	125	**40**	155	20
6	P50	125	50	155	20	0	0	0	0	125	**50**	155	20
7	P60	125	60	155	20	0	0	0	0	125	**60**	155	20
8	DAF sludge P40 (dairy processing)	125	0	155	20	7	**40**	1	1	132	**40**	156	21
9	Activated sludge P40 (dairy processing)	125	0	155	20	53	**40**	6	7	178	**40**	161	27
10	Cattle Slurry P40	125	23.5	155	20	80	**16.5**	116	10	205	**40**	271	30
11	Struvite1 P40 (potato wastewater processing)	125	0	155	20	19	**40**	4	0.0	144	**40**	159	20
12	Struvite2 P40 (sewage sludge processing)	125	0	155	20	20	**40**	0	0.0	145	**40**	155	20
13	Ash1 P40 (poultry litter processing)	125	0	155	20	0	**40**	77	22.2	125	**40**	232	42
14	Ash2 P40 (sewage sludge processing)	125	0	155	20	0	**40**	6	14.2	125	**40**	161	34
15	**Balanced N,P,K,S mineral**	**125**	**40**	**155**	**20**	0	**0**	0	0	125	**40**	155	20
16	DAF sludge P40 (dairy processing)	118	0	154	19	*7*	***40***	*1*	*1*	125	**40**	155	20
17	Activated sludge P40 (dairy processing)	72	0	149	13	*53*	***40***	*6*	*7*	125	**40**	155	20
18	Cattle Slurry P40 banalce	45	23.5	39	10	*80*	***16.5***	*116*	*10*	125	**40**	155	20
19	Struvite1 P40 (potato wastewater processing)	106	0	151	20	*19*	***40***	*4*	*0.0*	125	**40**	155	20
20	Struvite2 P40 (sewage sludge processing)	105	0	155	20	*20*	***40***	*0*	*0.0*	125	**40**	155	20
21	Ash1 P40 (poultry litter processing)	125	0	78	-2	*0*	***40***	*77*	*22.2*	125	**40**	155	20
22	Ash2 P40 (sewage sludge processing)	125	0	149	6	*0*	***40***	*6*	*14.2*	125	**40**	155	20

The trial was setup using a randomized block layout for P MFE and balance fertiliser plots respectively, with 5 replications for each treatment. The individual plots measured 2 m wide by 6 m long placed alongside each other with the spray lines used as a buffer strip. In between replicates 2 meter wide alley acting as a buffer. The spray used to mark out the trial was glyphosate. After application of the fertilizers it was observed that a high proportion of volunteer clover was emerging in the plots. Clovers along with other broadleaved species were controlled with an application of fluroxypyr-methyl in mid-May 2019.

RDFs were applied to the plots by hand spreading following a distribution of fertilisers as even as possible to cover each plot area. In order to get an even application RDFs were applied walking up and down the plots a minimum of 3 times. The mineral fertilisers applied using a barrow applicator (Figure 6). The barrow applies fertilisers at set rates at a 2 meter width. This was first calibrated to put out the correct amount and then walked behind up and down through each of the plots.

Figure 5: Mineral and RDF fertilizers weighing out facility before application to respective plots.

Figure 6: 2 m wide barrow fertilizer spreader for applying mineral fertilizer to relevant plots at pre -calibrated rates.

3. Monitoring and evaluation

3.1. Soil sampling and analysis

Soil samples were collected from the target site to assess the background soil quality and textural class. This was done by first splitting the trial site into two blocks from front to back and then sampling each block in a "W" shaped manor to accomplish a representative sample. Seven soil samples were collected in this method from each of the two blocks. The results from the textural analysis as shown in Figure 7 ("Pippette method", (ISO 11277, 1998)) showed the soil type to be a sandy loam.

To assess the soil quality and phosphorous build up from RDF applications, representative soil samples were collected from all experimental plots before application of the fertilisers and again after subsequent harvesting of grass. This was achieved using a 10 cm long by 1.5 cm wide (at the tip) soil corer. In order to achieve a representative sample from each plot, the plots must be sampled from lengthways from front to back in a "W" shaped approach. In order to achieve this, a minimum of 6 samples were taken. These samples must then be mixed in order to achieve a homogenous composite sample for each plot. The sample was then placed in a small cardboard box labeled with the plot number. This process was carried out for each individual plot. Samples were then dried in the oven at 40˚C for 72 hours. Once dried the samples were placed in a mechanical 2 mm soil sieving machine (Figure 8) where large amounts of debris are blown off and 2 mm dried sieved samples are collected (Figure 9).

Figure 7 Soil particle analysis. Sub samples are taken from the beakers immediately in order to calculate suspended solids.

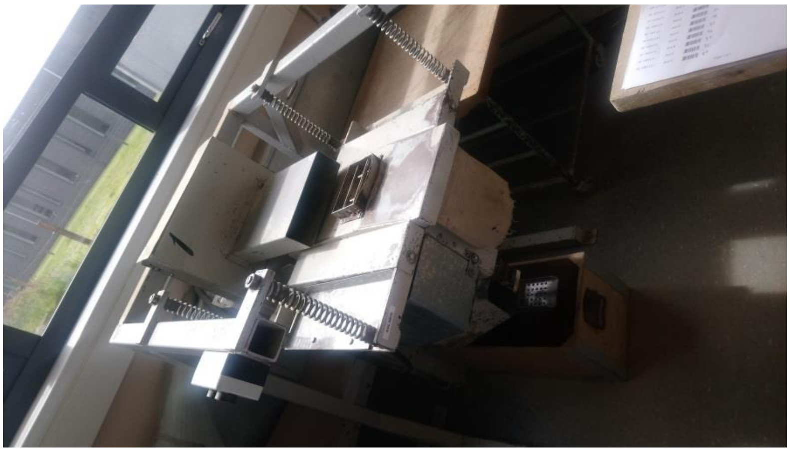

Figure 8 Mechanical 2 mm soil sieving facility.

Figure 9 Dried 2 mm sieved soil samples in cardboard box for nutrient and metal analysis.

Total carbon (TC) and total nitrogen (N) were measured by high temperature combustion method using LECO TruSpec CN analyser. The concentrations of nutrients (phosphorus (P), potassium (K), magnesium (Mg), sulphur (S), sodium (Na), and calcium (Ca) were determined by an Agilent 5100 synchronous vertical dual view inductively coupled plasma optical emission spectrometer (Agilent 5100 ICP-OES) following the microwave-assisted acid digestion of oven dried 2 mm sieved samples (USEPA, 1996) (Figure 10). Morgan's P and Mg were measured using a Lachet Flow through analyser. This is a colourmetric test. Morgans K was measured using a flame photometer. This is also a colourmetric test. Both lime requirement and pH were measured using a pH meter.

Figure 10 ICP-OES spectrometer facility to analyse soil samples for nutrients and metals.

3.2. Grass sampling and analysis

The plots were harvested in order to determine grass yields and quality by taking grass samples from each plot for analysis. Figure 11 shows grass growing pattern across control, RDF and mineral fertiliser plots. Plots were harvested by either a Haldrup trial plot grass harvester by Deutz Fahr or an Etesia commercial lawnmower (Figure 12). The plots were being harvested to simulate a silage season in Ireland. This means the first cut took place on the 24th of May after 6 weeks of fertiliser application. Figure 13 shows the first grass harvesting by Etesia trial plot mower. Subsequently, harvests will be taken place up to 4th cut for balance fertiliser application plots until October 2019 to simulate a situation where a farmer has let stock in to graze a paddock after last silage cut has taken place before the animals are housed for the winter. For P MFE trial plots, harvests may take place more than 4 cuts depending on the yield response and residual availability of P in the soil.

Both the Haldrup and Etesia cut at a height of 5 cm. The Etesia has a cut width of 1.24 m while the haldrup has a cut width of 1.5 m. The alleys and headlands in between and at the top and bottom of the plots were cut first to allow the harvester to run into the alleys from the plot and thus get an accurate harvest on the trial plots. Plots were harvested taking cuttings from the center. Harvested grass from each plot was weighed to calculate the yield and then sub-sampled in order for dry matter and further lab analysis to be carried out.

The subsamples are weighed and then dried in perforated plastic bags in an oven at 70˚C for 72 hours. Once dried the samples were grounded and sieved to 2 mm size and used for analysis. Total crop P, K, S, Mg, Mn, and Zn were all analysed using ICP-OES spectrometer. Total N and C were analysed using a combustion analyser (LECO TruSpec CN analyser).

Figure 11 Grass growth in zero fertiliser plot (less dense sward indicating a control plot receiving no fertilizer) and recycling *vs.* synthetic fertiliser plots.

Figure 12 Etesia trial plot mower with a 1.24 m cutting width and 5cm cutting height (left) and a Haldrup combine harvester (right).

Figure 13 First harvest of grass in-progress using the Etesia with noticeable 1.24 m cutting width out of each 2 m wide plot.

4. Demonstration process

4.1. Open day

The demonstration trial of the use of RDFs at Teagasc Johnstown Castle agronomic grassland plots was presented at two open days – 1) Teagasc DairyBEEF2019 Open

Day, held on 21 May 2019, and 2) Teagasc Crops & Spreaders Open Day, held on 26 June 2019. The ReNu2Farm project members from Teagasc, Institute of Technology Carlow, Cork Institute of Technology (CIT) and University of Limerick (UL) attended the open days (Figure 14, Figure 15) (IT Carlow), and presented project scope and technical information about the demonstration trial to a wide range of participants e.g. farmers, Teagasc nutrient and fertiliser management advisors, interdisciplinary researchers within agri-environmental science, agricultural contractors and consultants (Figure 16, Figure 17).

Teagasc Johnstown Castle hosted the DairyBEEF Open day on the 21st of May 2019. For the open day a board poster was made detailing the aim, objectives, and work involved as part of Teagasc collaboration and deliverable for ReNu2Farm project. Researchers (see Figure 14) from Teagasc, IT Carlow, CIT and UL represented the ReNu2Farm stance during the open day to demonstrate findings, and answer questions from farmers, entrepreneurs, and advisors (Figure 16, Figure 17). The trial site was also represented by cutting off the discards and alleys and putting signs detailing the treatments and fertilizer rates for each individual plot (Figure 18, Figure 19). A survey questionnaire was administered and conducted for the open day to be completed by farmers, advisors, and agricultural entrepreneurs. Technologies to recover nutrients and produce RDFs are available on the market, but until now they have remained little-used by farmers. Within the ReNu2Farm project, more specifically with this survey, we would like to gain information on the current knowledge and existing barriers on the use of RDF.

Table 2 Total N, P, K and S levels in RDFs fertiliser products.

Recycling Derived Fertilisers (RDFs)		Total nutrient (dry weight basis)			
	DM (%)	N (%)	P (%)	K (%)	S (%)
Cattle Slurry	9.0	3.3	0.6	4.3	0.4
Dairy sludge (Activated brown)	12.2	4.5	3.4	0.5	0.6
Dairy sludge (lime treated DAF)	25.0	2.0	10.7	0.4	0.3
Struvite from Potato processing	58.3	5.1	10.7	1.2	0
Struvite from Sewage sludge	51.3	5.1	10.0	0.1	0
Ash from Poultry litter	100	0.02	5.5	10.7	3.1
Ash from Sewage sludge	100	0.03	8.4	1.3	3.0

Groups of the public/participants gathered during the open day at the ReNu2Farm board stand and were taken down to the plots for a demonstration and talk (Figure 19). This was done throughout the day in order to show as many people as possible the benefits of using RDFs compared to synthetic fertilisers. The aspects of the talk involved demonstrating the differences in sward thicknesses between different plots, showing people the RDF product samples, and educating the public on the manufacturing and nutrient content of the RDFs (Table 2 shows nutrient content of RDF samples). The main focus of this trial demonstration was dissemination, education, and knowledge transfer.

Figure 14 ReNu2Farm researchers from Teagasc (Patrick Forrestal, Cathal Redmond, SM Ashekuzzaman), Institute of Technology Carlow (Thomae Kakouli-Duarte, Anna Karpinska), Cork Institute of Technology (Ciaran O'Donnell) and University of Limerick (Achim Schmalenberger, Lea) during DairyBEEF2019 Open Day at Teagasc Johnstown Castle, Co. Wexford.

Figure 15 ReNu2Farm researcher from Teagasc (Patrick Forrestal, Cathal Redmond, SM Ashekuzzaman, John B Murphy, Martin Bourke), Institute of Technology Carlow (Thomae Kakouli-Duarte, Anna Karpinska) during Teagasc Crops & Spreaders Open Day at Teagasc Oak Park on 26 June 2019.

Figure 16 ReNu2Farm researchers interacted with public and farming community during Teagasc Crops & Spreaders Open Day on 26 June 2019.

Figure 17 Public and farming community being engaged by the ReNu2Farm researchers from the University of Limerick, Carlow Institute of Technology, Institute of Technology Cork, and Teagasc during DairyBEEF2019 Open Day.

Figure 18 Agronomic trial and demonstration site with signs detailing fertilizer applications per plot located at Teagasc Johnstown Castle dairy grassland.

Figure 19 Members of the public and farming community being engaged by Teagasc researcher during DairyBEEF2019 Open Day.

4.2. Field visit and demonstration

A field visit was organized by Teagasc ReNu2Farm researchers on 20th June 2019 to engage Teagasc Environment Research Centre research/advisory staffs and students with on-going agronomic trial using RDF products (Figure 20, Figure 21). The purpose of this demonstration was to provide a better knowledge transfer (including benefits and characterization of RDF samples) on using different bio-based recycling products for fertiliser value under EU funded ReNu2Farm and aligned project.

Figure 20 Teagasc researcher SM Ashekuzzaman engaging with members from Teagasc Johnstown Castle Environment Research Centre at the agronomic field trial site.

Figure 21 Members from Teagasc Johnstown Castle Environment Research Centre during field visit at the ReNu2Farm agronomic demonstration trial site.

4.3. Seminar and Conference

Researchers from Teagasc and IT Carlow attended the Irish Plant Scientists' Association Meeting (IPSAM2019) on 25 – 27 June at IT Carlow, Ireland to disseminate knowledge (ReNu2Farm findings and its impact for soil nutrient sustainability) from ReNu2Farm and aligned projects (Figure 22). Results from agronomic field trial (poster presentation, Figure 23) indicated that balanced application of RDF products (listed in Table 2) can achieve similar grass production to what mineral-based chemical fertilisers provide. This has great potential to reduce and replace the use of mineral fertilisers and saving €.

Figure 22 Teagasc and IT Carlow ReNu2Farm researchers at the Irish Plant Scientists' Association Meeting (IPSAM2019), IT Carlow, Ireland.

Figure 23 Poster presentation entitled "Using recycling-based fertilisers towards better crop nutrient stewardship within agro-industrial food supply chain" by the Teagasc researcher SM Ashekuzzaman at the IPSAM2019, 25 – 27 June, IT Carlow, Ireland.

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