ADVANCES IN »
HYDROTHERMAL CONVERSION OF INDUSTRIAL BIOGENIC RESIDUES INTO INTERMEDIATE BIOENERGY CARRIERS

RESULTS FROM THE **F-CUBED PROJECT**

F-CUBED

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 884226.

NPK recovery potential from TORWASH® treated biomass

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Principle

Paper bio-sludge

Olive pomace

Orange peels

Torwash®

N P (K)

TORWASH solids

TORWASH effluent

Nutrient recovery

Struvite

$(NH_4)MgPO_4*6H_20$

Characteristics

- Elemental composition of TORWASH® **dewatered solids** (filter press cake)

			Paper sludge		Waste olive pomace		Orange peels
			10.09.21	21.09.21	11.11.21	12.11.21	29.04.22
macronutrients	C	%	46.2	46.8	66.1	65.3	56.1
	N	%	4.3	4.6	1.5	1.5	2.2
	P	mg/g	21.2	23.4	0.2	0.2	0.2
	K	mg/g	0.9	0.5	1.1	1.4	1.2
	Ca	mg/g	21.3	22.2	4.3	4.5	11.2
	Mg	mg/g	3.0	2.7	0.1	0.1	0.2
	S	mg/g	8.8	9.4	1.2	1.0	1.4
micronutrients	Fe	mg/g	6.7	6.8	0.2	0.2	0.4
	Mn	mg/kg	11500	14400	2.78	1.95	3.63
	Zn	mg/kg	1125	1199	26.1	19.7	12.9
	Cu	mg/kg	64.9	65.6	37.0	42.2	21.0
	Mo	mg/kg	7.97	10.7	0.55	0.53	0.25
	Ni	mg/kg	10.5	9.3	1.45	1.14	3.95
heavy metals	Cd	mg/kg	11.2	10.2	<0.025	0.032	<0.100
	Cr	mg/kg	32.3	32.8	3.97	1.67	2.31
	Pb	mg/kg	39.3	38.7	0.282	0.272	3.03
	V	mg/kg	22.2	23.3	0.408	0.500	<0.100
	As	mg/kg	3.16	3.13	<0.025	<0.025	<0.100
	Hg	mg/kg	0.135	0.143	<0.025	<0.025	<0.100

Characteristics

- Elemental composition of TORWASH® **liquid effluent**

			paper sludge		waste olive pomace		orange peels
			08.09.21	21.09.21	11.11.21	12.11.21	25.04.22
	pH		6.3	6.1	4.4	4.4	4.1
macronutrients	TNb	mg/L	900	1060	142	127	291
	NH_4-N	mg/L	244	177	11.8	8.5	21.4
	P	mg/L	81.1	38.2	85.7	87.8	32.7
	K	mg/L	104	81.0	1590	1672	481
	Ca	mg/L	60.1	74.0	73.3	49.8	144
	Mg	mg/L	48.8	24.5	76.9	64.0	48.9
	S	mg/L	313	243	44.8	45.2	33.1
micronutrients	Fe	mg/L	12.1	4.2	0.6	0.7	0.6
	Mn	µg/L	16400	21100	930	560	41
	Zn	µg/L	186	167	1210	790	<5.0
	Cu	µg/L	2.5	1.7	5.54	3.12	67
	Mo	µg/L	45.0	63.0	<0.50	<0.50	<5.0
	Ni	µg/L	37.0	34.0	9.97	10.9	6.0
heavy metals	Cd	µg/L	<1.0	<1.0	<0.50	<0.50	<5.0
	Cr	µg/L	32.0	24.0	2.78	2.4	<5.0
	Pb	µg/L	1.3	<1.0	5.97	4.38	<5.0
	V	µg/L	13.0	12.0	<0.50	<0.50	<5.0
	As	µg/L	24.0	25.0	2.3	3.1	<5.0
	Hg	µg/L	0.076	0.092	<0.25	<0.25	<5.0

Characteristics

- Organic compounds in TORWASH® **liquid effluent**

		paper sludge		waste olive pomace		orange peels
		08.09.21	21.09.21	11.11.21	12.11.21	25.04.22
COD	g/LO_2	9.8	7.9	27.9	27.8	43.4
Sucrose	mg/L	1590	-	650	618	320
Glyceraldehyde	mg/L	166	156	563	546	379
Levulinic acid	mg/L	175	123	152	149	62.0
Furfuryl alcohol	mg/L	252	323	249	300	164
1,2,4-Benzenetriol	mg/L	-	56.0	170	215	50.0
Hydroxymethylfurfural	mg/L	64.5	99.3	294	205	218
Furfural	mg/L	42.7	187	307	346	140
Glucose/Galacturans	mg/L	1090	1360	440	201	1240
Fructose (Malic acid)	mg/L	201	214	1310	922	-
Pyruvaldehyde	mg/L	93.8	46.0	582	474	109
Formaldehyde/ (Dihydroxyacetone)	mg/L	-	97.4	447	428	617
Formic acid	mg/L	356	84.6	1290	1540	772
Acetic acid	mg/L	298	371	2210	2580	663
MeOH	mg/L	-	-	1510	1540	898
Acetaldehyde	mg/L	-	257	-	-	-
Et/OH (Benzoic acid)	mg/L	510	272	1640	2600	5200
Resorcinol	mg/L	112	79.1	na	na	196
Catechol	mg/L	134	56.4	na	na	12.3
Phenol	mg/L	3.40	-	na	na	15.8
PAH*	µg/L	na	na	bql**	bql**	bgl**

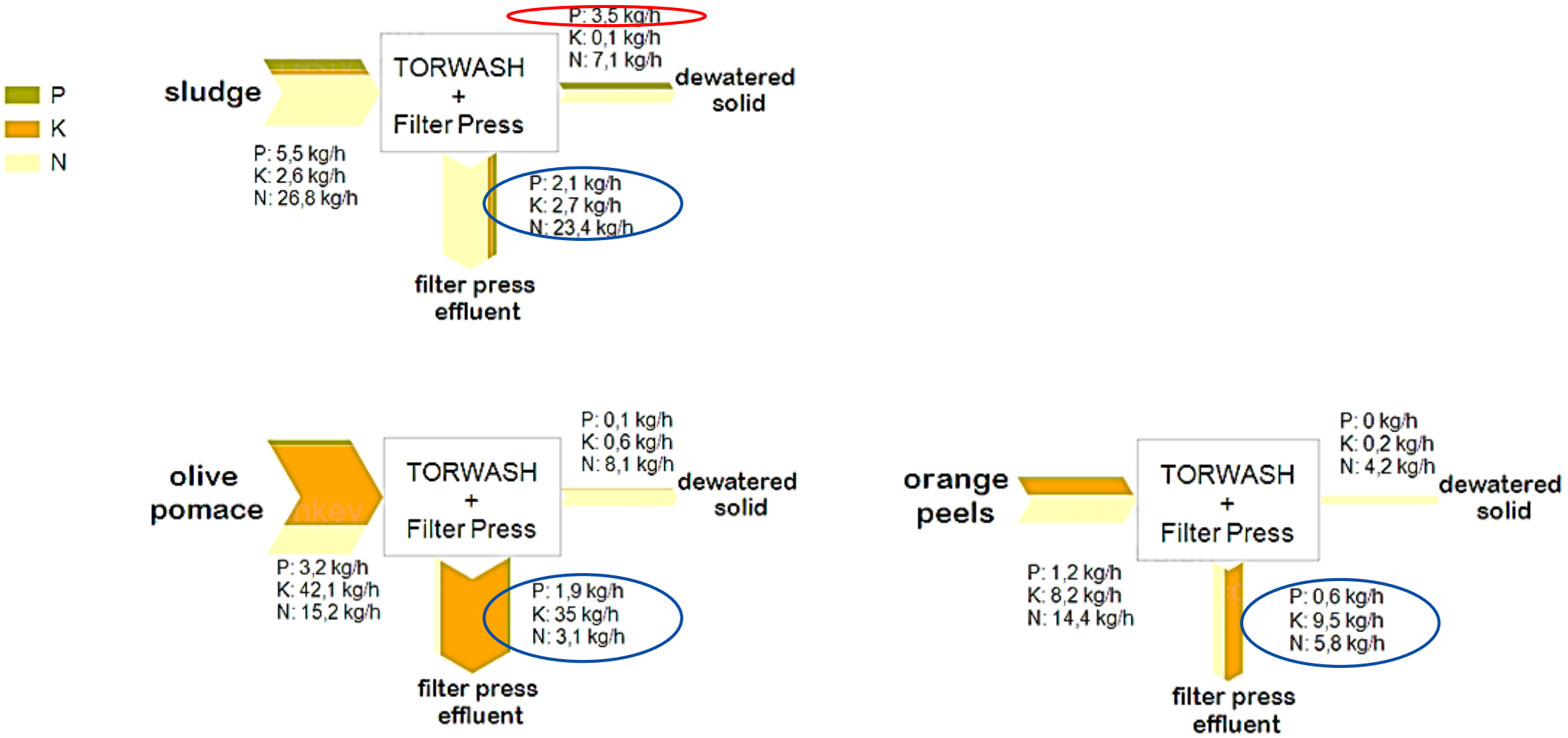
*PAH (sum of 16 polycyclic aromatic hydrocarbons) as organic pollutants; **content of all of the polycyclic aromatic hydrocarbons analyzed was less than quantification limit
na=not analyzed

Characteristics

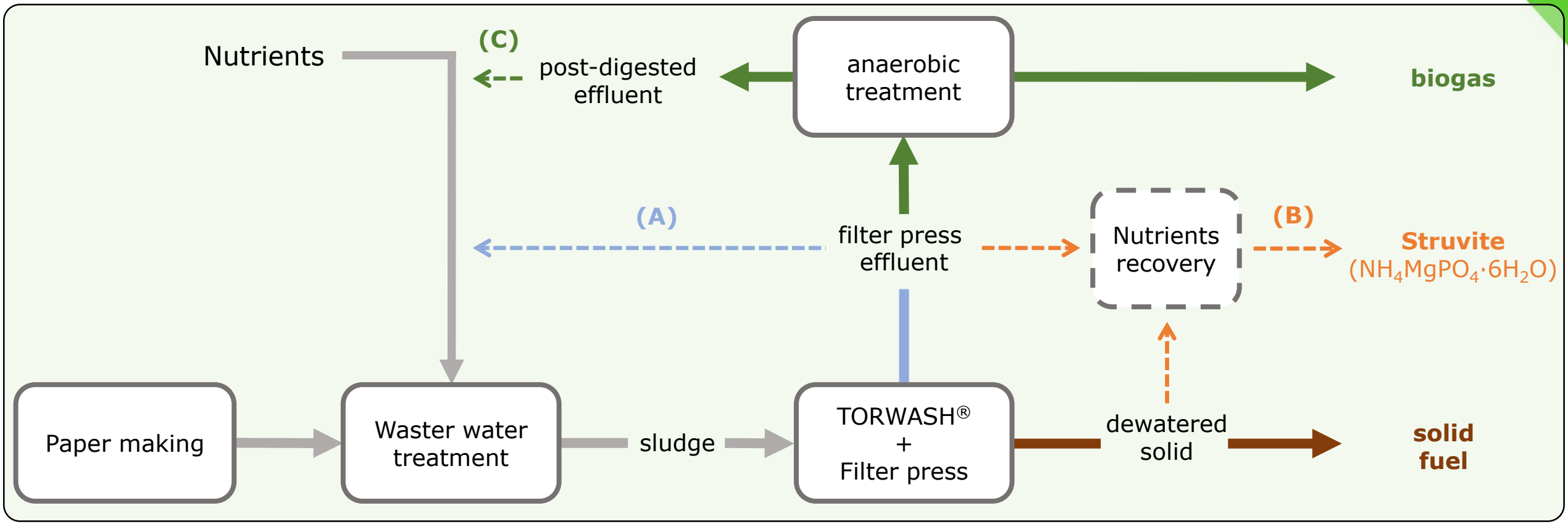
- Post-digested **liquid effluent**

			paper sludge		waste olive pomace		orange peels	
				x 2.25		x 8.4		x 12.5
	pH		8.1		7.5		7.5	
macro-nutrients	TNb	mg/L	412	927	103	862	49.4	618
	NH_4-N	mg/L	317	713	75.3	632	31.2	390
	P	mg/L	23.9	54	39.9	335	4.7	59
	K	mg/L	40.2	90.5	311	2610	50.9	636

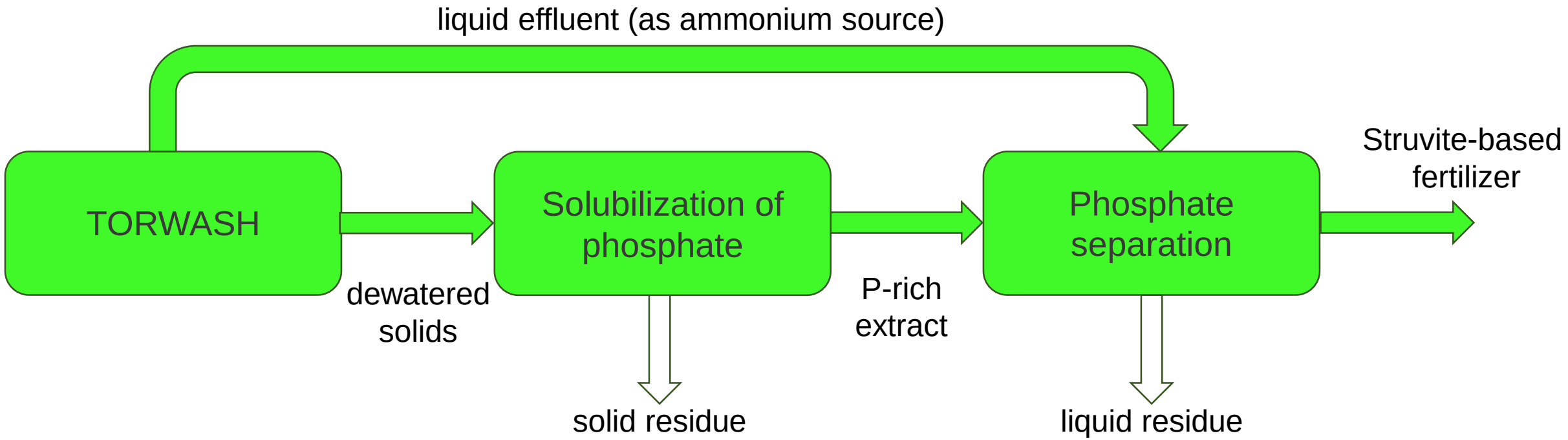
NPK mass flows



Potential of nutrient recovery from paper sludge



(B) Potential of nutrient recovery from paper sludge



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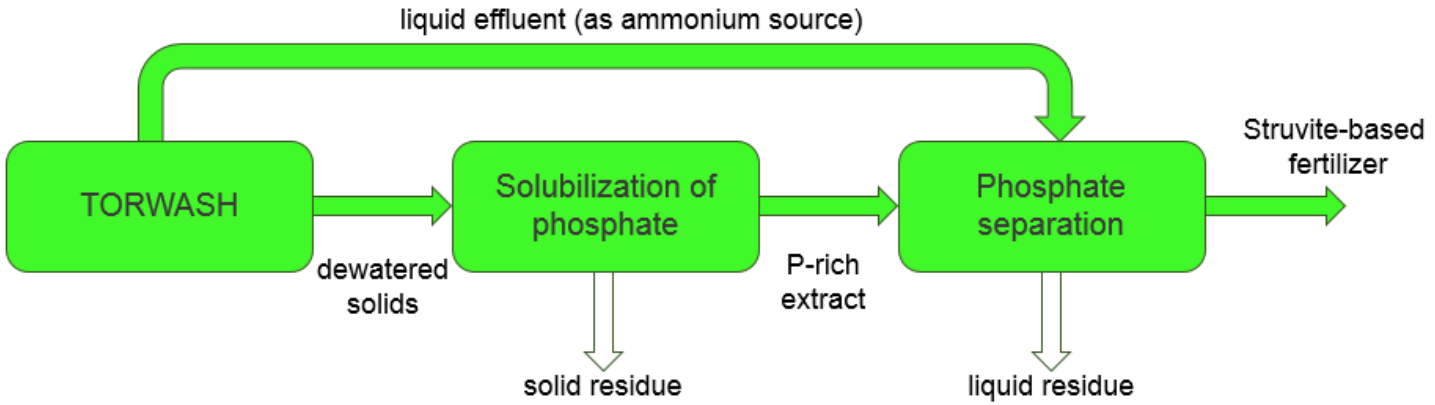
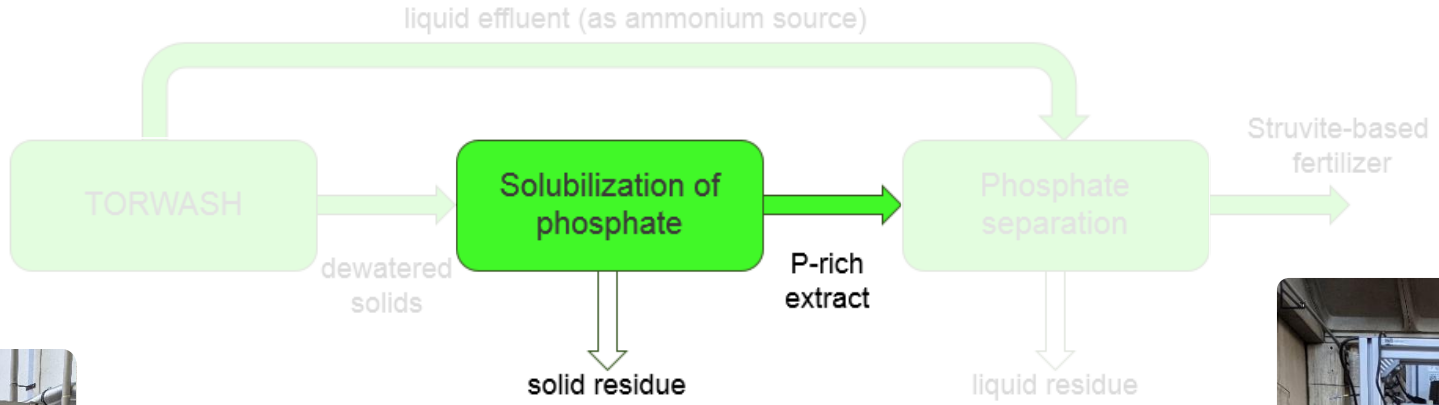
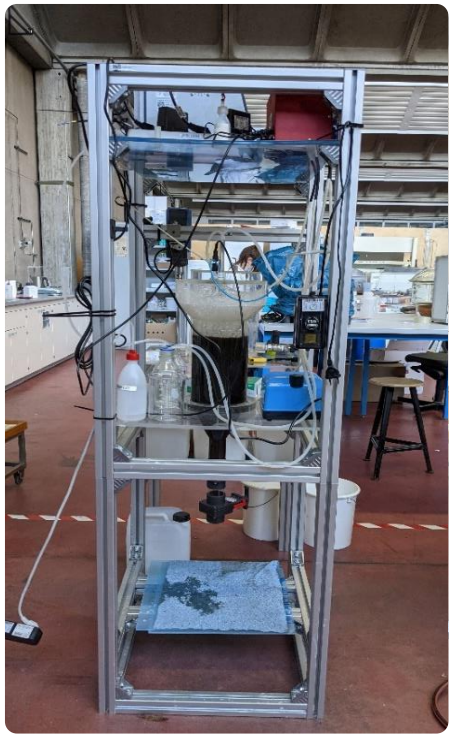


(B) Potential of nutrient recovery from paper sludge

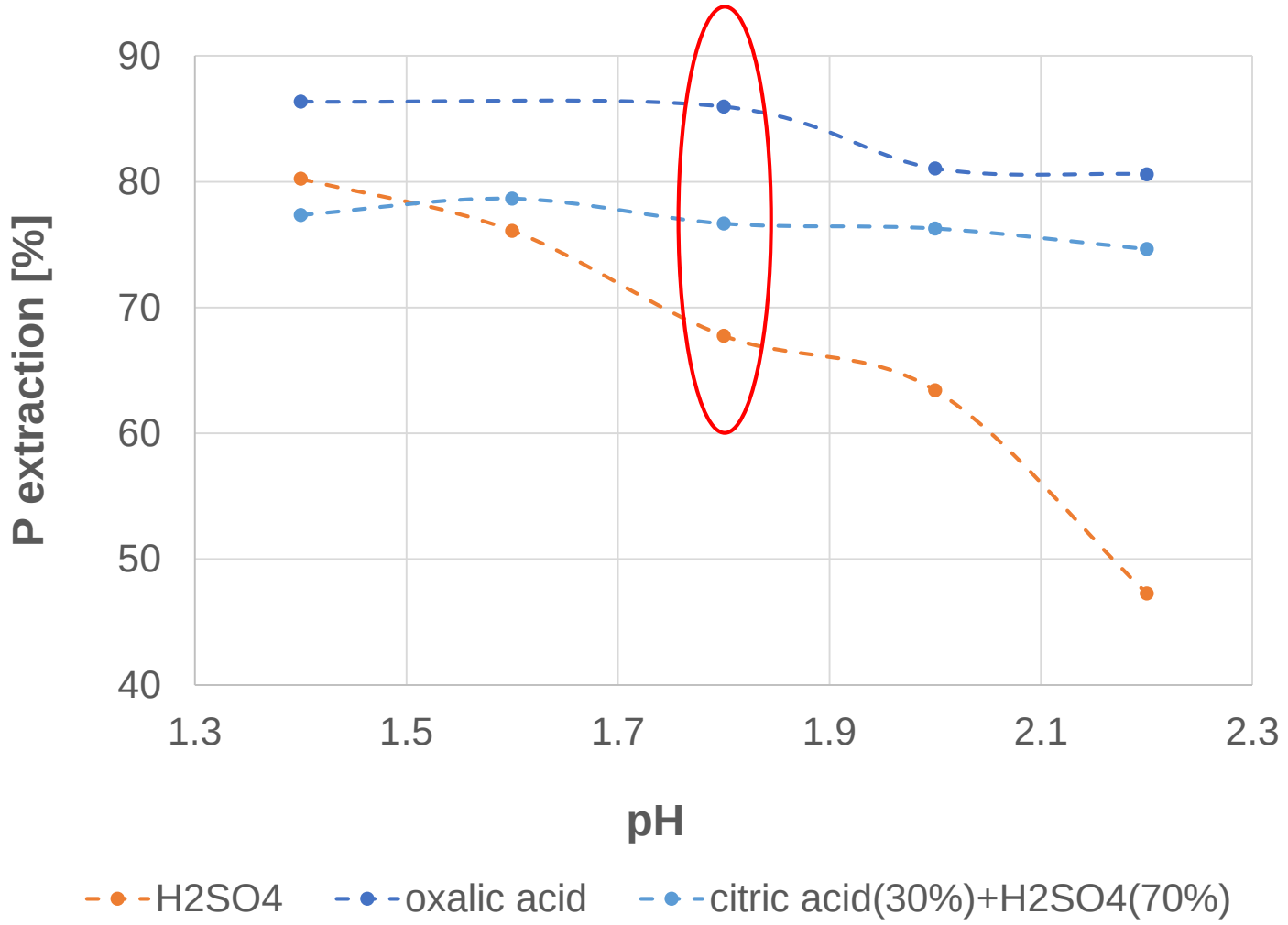


V = 8 L



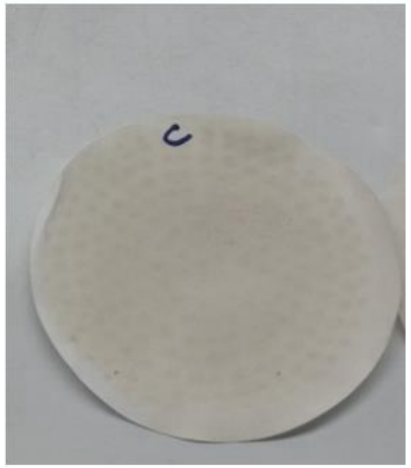
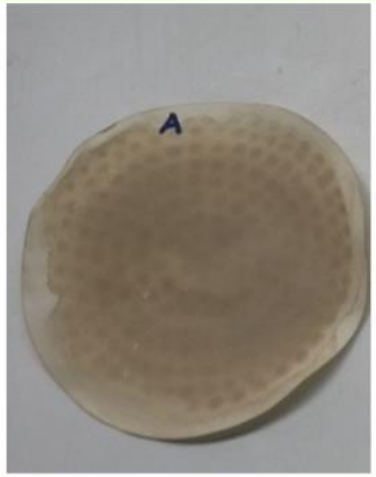
V = 5 L

Phosphate solubility test



$$P\ extraction, [\%] = \frac{amount\ of\ P\ in\ the\ dewaterd\ solids - amount\ of\ P\ in\ the\ solid\ residue}{amount\ of\ P\ in\ the\ dewatered\ solids} \cdot 100$$

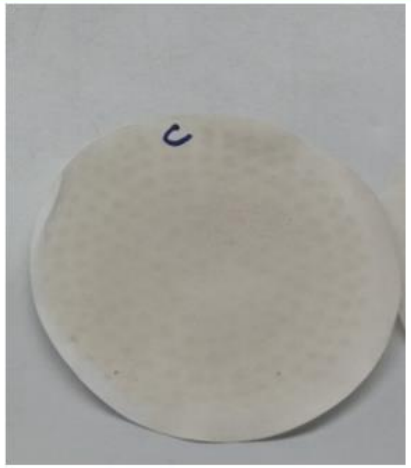
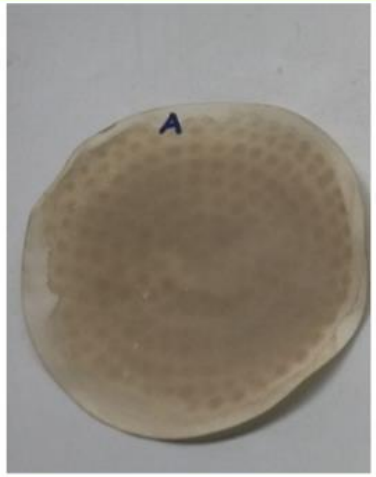
Phosphate solubility test

Lab scale		Mini-plant
		
from P-rich extract produced with citric and sulphuric acid	from P-rich extract produced with oxalic acid	

acid	precipitate yield	precipitate purity	direct use precipitate as fertilizer	P recovery	NH4-N recovery
Oxalic acid	+	0	0	+	0
Citric+H_2SO_4	-	+	+	0	0

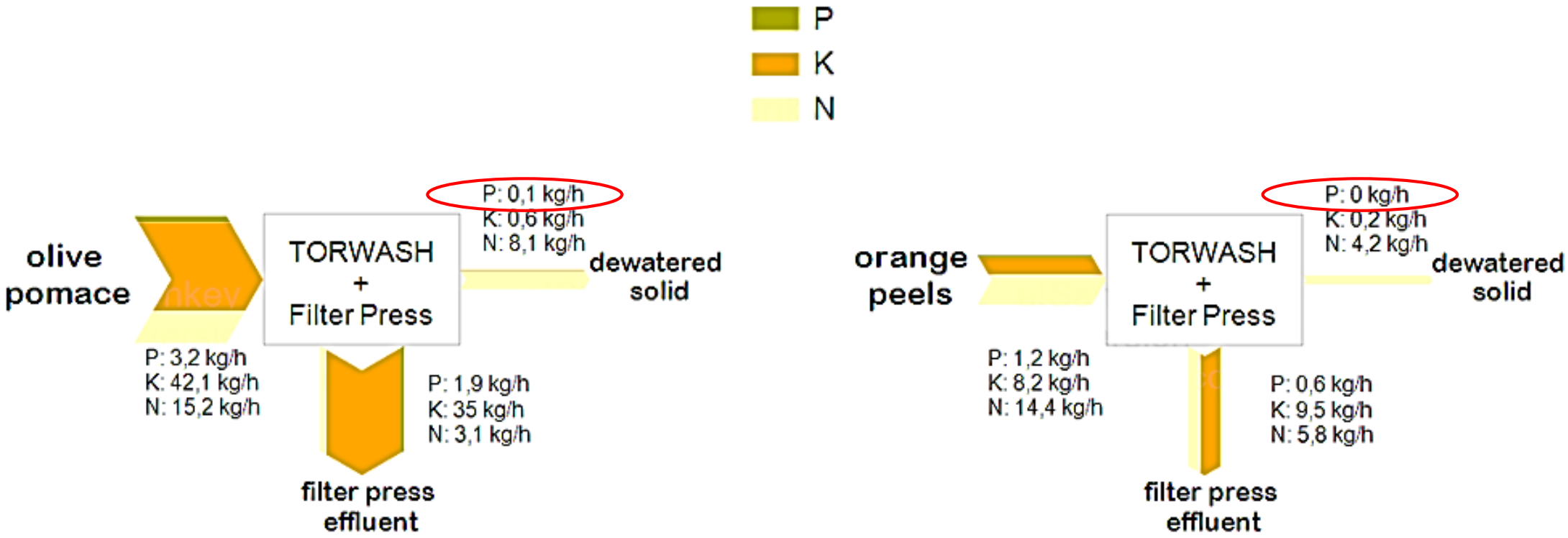
+, 0, - are performance indicators by the subjective perception of the author ranging from positive, neutral to negative respectively

Phosphate solubility test

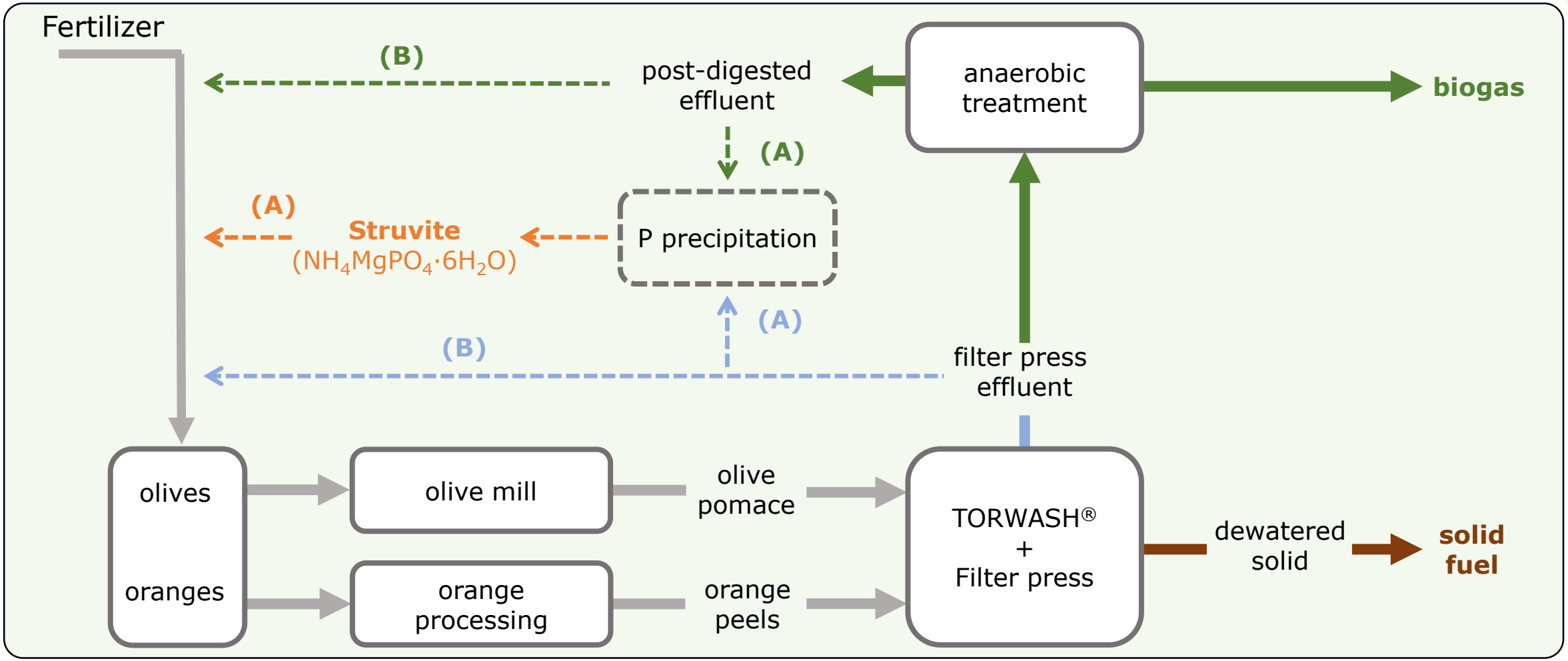
Lab scale		Mini-plant
		
from P-rich extract produced with citric and sulphuric acid	from P-rich extract produced with oxalic acid	

- Total P recovery in miniplant: 38%
- Design of extraction unit needs to be optimized:
  - Mixing conditons
  - Improved wetting and resuspension of dry filter press cakes during acid leaching

Potential of nutrient recovery for feedstocks with low P content



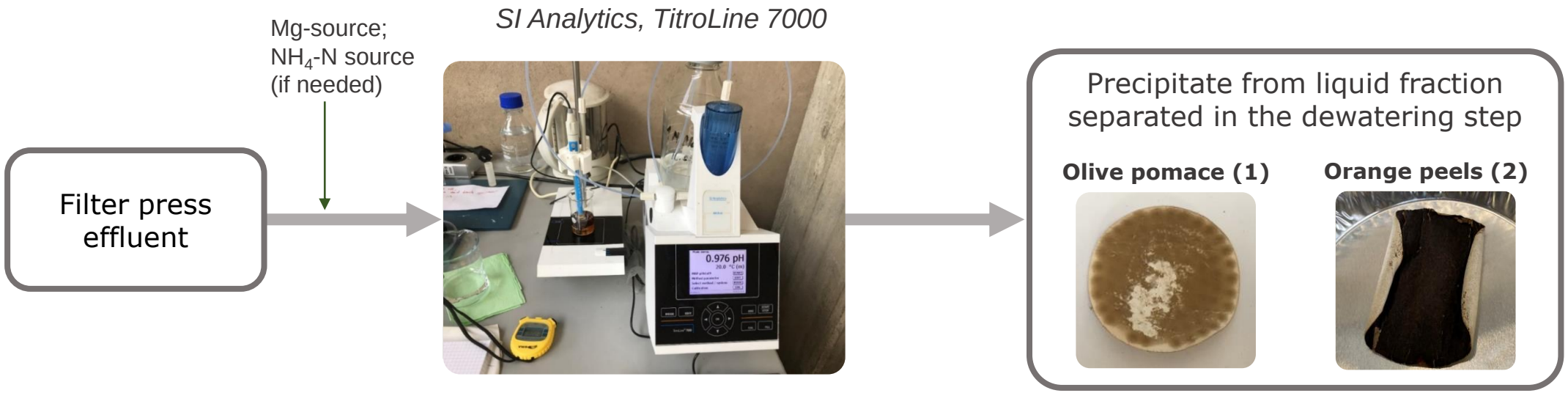
Potential of nutrient recovery for feedstocks with low P content



(A) Recovery of P and part of N (as ammonium) in the form of a ready-to-use fertilizer

(B) Recycling effluents as liquid fertilizer

Potential of nutrient recovery for feedstocks with low P content



Operating parameters		**1**	**2**
Adding Mg-source		yes	no
Adding NH_4-N-source		yes	no
Initial NH_4-N:Mg:P	mol/mol/mol	1.8 : 1.6 : 1	1.4 : 1.9 : 1
Reaction time	min	60	60
pH	-	9.0	9.0
Performances			
Precipitation yield	g/L	0.5	0.5
P recovery form the solution	%	50	60
NH_4-N recovery from the solution	%	13	15

Potential of nutrient recovery for feedstocks with low P content

Filter press effluent

Mg-source;
NH_4-N source
(if needed)

SI Analytics, TitroLine 7000

Precipitate from liquid fraction separated in the dewatering step

Olive pomace (1)

Orange peels (2)

effluent	P content	Balanced content NH_4-N:P	Balanced content Mg:P	NaOH consumption	P recovery	Precipitate purity
Olive pomace	-	-	0	-	0	0
Orange peels	-	+	+	0	0	-

+, 0, - are performance indicators by the subjective perception of the author ranging from positive, neutral to negative respectively

Conclusion

- Feedstocks provide specific NPK contents, mobilities, availabilities

  → different approaches for recovery

Conclusion

- Paper sludge
  - Limited knowledge for direct integration of nutrient recovery to the WWT process
  - Production of a struvite based mineral fertilizer is expected to be feasible

Conclusion

- Torwash® process provided good separation of nutrients **from orange peels** and **olive pomace** to the effluent

  → Immediate struvite precipitation is an option

Outlook

- Paper sludge
  - Further development of the up-scaled process
  - Fertilizing tests (lab & field)
  - Assessment of feasibility, cost effectiveness and additional benefits (especially for direct nutrient recovery to the WWT process)

THANK YOU

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