MINISTRY OF THE ENVIRONMENT, CONSERVATION AND PARKS (MECP) and MINISTRY OF AGRICULTURE, FOOD AND AGRIBUSINESS (MAFA)

Characteristics of Digestate from Various Ontario Anaerobic Digester Systems

Preliminary Findings for Collaborative Study by MECP and MAFA

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Overview – AD Study

Preliminary Findings of the Collaborative AD Study by MECP and MAFA

- 1. Introduction Update on Ontario AD Study
 - Context, Design, and Scope
 - Sample Collection and Handling
 - Analytical Parameters
 - AD Facility Details
- 2. Preliminary Observations
- 3. Preliminary AD Study Highlights
- 4. Acknowledgements
- 5. Appendices





Introduction – Context, Design and Scope

Ontario Context (Study initiated 2020; re-launched 2023)

- Significant growth in biogas sector of Ontario organics industry
- July 2021 amendments to Nutrient Management (NM) Regulation (O. Reg. 267/03), allow farms to receive more off-farm materials and source-separated organics (SSO)
- Anaerobic digestate is land applied in Ontario as a beneficial nutrient amendment; it supports soil health and crop growth



- Study focused on science gaps to better characterize the quality of digestates applied in Ontario
- Outcome/aim to better inform management practices for recycled organic residuals in Ontario

Study Scope / Design

- Voluntary participation by 9 facilities; 4 sampling events over 1 year; conducted by MECP and MAFA
- Representation of various anaerobic digester systems across Ontario:
 - On-farm vs. Off-farm
 - Managed by permit (e.g., environmental compliance approval) vs. under the Nutrient Management regulation
 - Feedstocks processed food waste and agricultural materials vs. municipal SSO



Introduction – Digesters, Sampling, and Analytical Parameters

Sample Collection and Handling

- Three types of digestate samples were collected:
 - Whole: As-is digestate, slurry fresh from digester (not long-term storage)
 - Separated Liquid: Liquid fraction from post-digestion solid-liquid separation
 - Separated Solid: Solid fraction from post-digestion solid-liquid separation
- Submitted to MECP & Agriculture and Agri-Food Canada (AAFC), Commercial lab, and Academic research lab.

Analytical Parameters

- Nutrients, metals, pathogens*
- Emerging contaminants (e.g., PFAS)*
- Foreign matter and plastics*
- Legacy organics (e.g., PCBs, pesticides)
- Microplastics (method development research ongoing)

Digesters - grouped based on predominant feedstocks

- Agri-Food Digesters that receive primarily manure, agricultural, and food processing feedstocks (4 facilities, represents ~12% of Ontario agri-food digesters)
- SSO Digesters that primarily receive municipal SSO (5 facilities, represents ~60% of Ontario SSO digesters)









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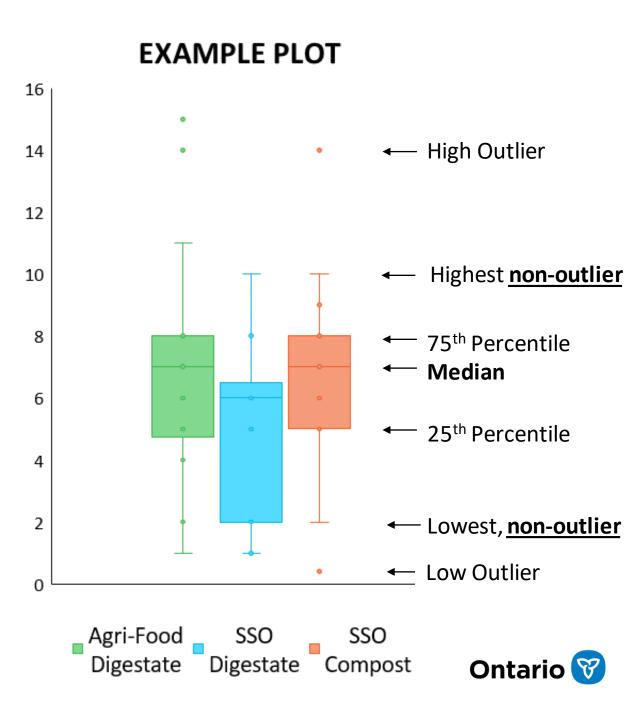
* Parameters included in today's preliminary presentation

Introduction – AD Facility Details

Facili	ty Code	Retention Time	Feedstock Proportions	Types of Feedstocks					
SIS	Α	160 days	69% on-farm 31% off-farm	Manure; plant residues; culled greenhouse crops; FOG; sugar; brewery byproducts					
igeste	В	50 days	65% on-farm 35% off-farm	Manure; feed mill residues; DAF; food processing residues; paunch manure					
Agri-Food Digesters	С	60-80 days	50% on-farm 50% off-farm	Manure; tree fruit residues; field crop residues; feed mill residues; FOG; DAF food preparation & distribution residues; ethanol production byproducts					
Agri	D	60 days	12% on-farm 88% off-farm	Manure; field crop residues; food processing residues; FOG; food preparation & distribution residues; cheese production byproducts					
	Е	50 days	50% SSO 50% on-farm	SSO; Greenhouse crop residues; vegetable processing residues					
Digesters	F	40 days	50% SSO 50% other	SSO; Pre-processed food preparation & distribution residues; food processing residues					
SSO Dige	G	40 days	50% SSO 50% other	SSO; FOG; food preparation & distribution residues					
	н	21 days	55% SSO 45% other	SSO; Food processing residues; food preparation & distribution residues; FOG; DAF; paunch manure					
	I.	15 days	100% SSO	SSO					

Preliminary Observations

- Data shown as "box and whisker" plots
- Facilities are grouped by primary feedstocks received/processed
- Plots show descriptive statistics on aggregated data, comparative statistics not completed
- Final analyses pending for Winter 2024 samples (75–100% of each dataset represented, depending on parameter)
- Digestate results qualitatively compared to compost dataset from MECP study completed in 2011



Regulated Metals

- NM Regulation regulates 11 metals in off-farm anaerobic digestion (AD) feedstock materials and *non-agricultural source materials* (NASM).
 - Two metals thresholds: "CM1" (eq. to Cat. AA compost) and "CM2" (the metals limit for land application as a NASM)
- Metals are limited as they may persist and accumulate in agricultural soils; can result in human or ecotoxic effects:
 - Arsenic (As), Cadmium (Cd), Mercury (Hg) and Lead (Pb) have negative human health and ecotoxic effects and no known benefits as micronutrients.
 - Chromium (Cr), Cobalt (Co), Copper (Cu), Molybdenum (Mo), Nickel (Ni), Selenium (Se), and Zinc (Zn) are essential micronutrients but can have negative effects at elevated levels.
 - The latter may be used as supplements for livestock or digester health.
- Metals are regulated and reported here on a dry weight basis, rather than "fresh" or "wet weight"
- See the Appendix for a full tabular summary of the aggregated metals data







Regulated Metals (As, Cd, Hg, Pb)

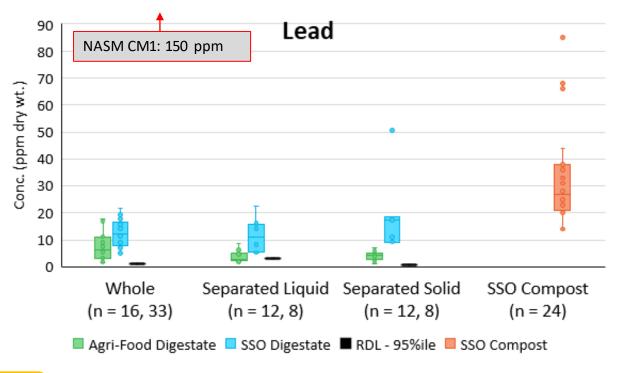
Comparison Between Agri-Food and SSO Digestate

- Levels of As, Cd, Hg, and Pb below NASM CM1 and As, Cd, and Hg mostly below detection.
- Agri-food and SSO digestate similar except for Pb, which is generally higher in SSO digesters although still well below CM1.

Comparison Between Compost and Digestate

- As, Cd, and Hg: Digestate comparable to SSO compost.
- Pb: Digestate generally lower than SSO compost.

Highlight: As, Cd, Hg, Pb very low in sampled digestates.

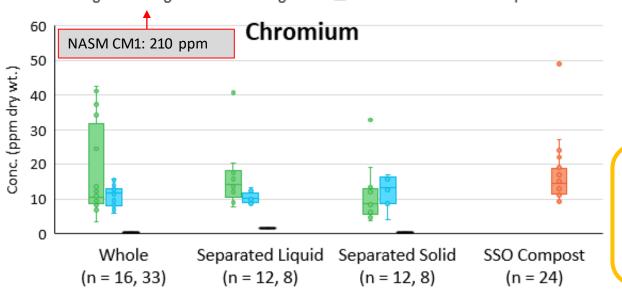


*RDL denotes laboratory Reported Detection Limit for samples analysed.

Regulated metals in Digestate (95th Percentile) compared to compost and standards. Values in ppm dry weight.

Analyte All values in ppm dry weight	Agri-Food Digestate: Whole (n = 16)	Agri-Food Digestate: Sep. Liquid (n = 12)	Agri-Food Digestate: Sep. Solid (n = 12)	SSO Digestate: Whole (n = 33)	SSO Digestate: Sep. Liquid (n = 8)	SSO Digestate: Sep. Solid (n = 7)	SSO Compost MECP 2011 Study (n = 24)	NASM CM1
Arsenic	<7.1	<5	<7.3	<10	<5	<5	8.7	13
Cadmium	0.5	<0.4	<0.1	<0.2	1.3	<0.1	0.6	3
Mercury	<0.2	<0.3	<0	0.1	<0.3	0.1	0.1	0.8

Regulated Metals (Cr, Cu, Zn)



400 Copper 0 350 Conc. (ppm dry wt.) 300 250 200 150 NASM CM1: 100 ppm 100 50 0 Whole Separated Liquid Separated Solid SSO Compost 9 (n = 12, 8) (n = 16, 33) (n = 12, 8) (n = 24)

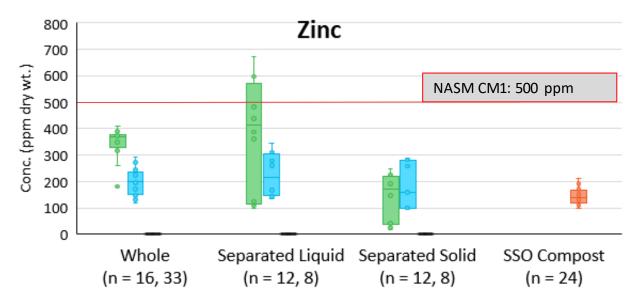
Comparison Between Agri-Food and SSO Digestate

- All results for Cr are below NASM CM1.
- Some Cu (many) and Zn (few) results exceed CM1 but not CM2.
- Cr, Cu, Zn in agri-food digestate often higher than SSO, likely due to nutritional additives in feed found in manure feedstocks.

Comparison Between Compost and Digestate

 Cr, Cu, and Zn: SSO digestates comparable to SSO compost. Agri-Food digestates are generally higher.

Highlights: Cu and Zn in sampled digestates sometimes exceed CM1, likely due to nutritional additives to livestock feed found in manure feedstocks. Values are still below Category A compost limits and well below CM2.

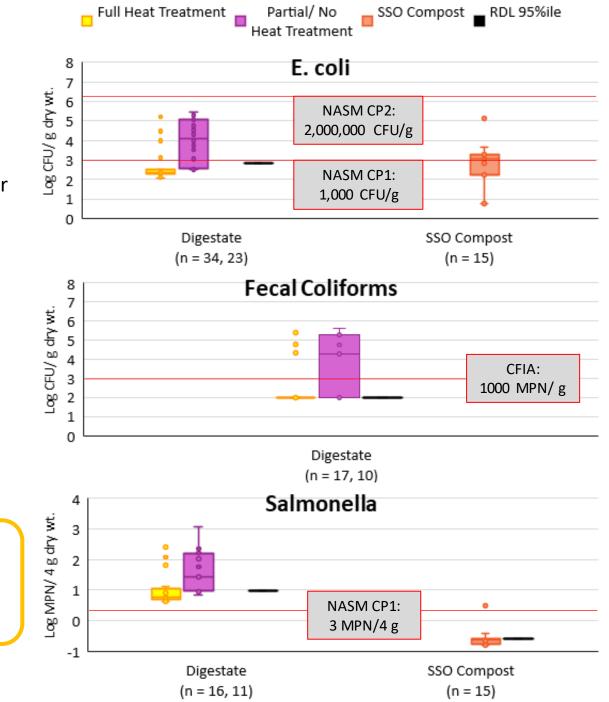


🔲 Agri-Food Digestate 📃 SSO Digestate 🔳 RDL - 95%ile 📕 SSO Compost

Regulated Pathogens & Indicators

- Cannot compare agri-food and SSO digestate due to variation in heat treatment regimes between facilities
- Facilities compared based on level of feedstock heat treatment:
 - Full Heat Treatment: all feedstocks treated at ≥50 °C for ≥20 hours or ≥70 °C for ≥1 hour
 - Partial/ No Heat Treatment of feedstocks.
- E. coli and fecal coliforms
 - All E. coli results below the NASM CP2 standard.
 - Full Heat-Treatment: all below CP1 and CFIA safety standards except for few outliers
 - Partial/ No Heat Treatment: most exceed CP1 and CFIA
- Salmonella
 - Full Heat-Treatment: all below detection limit (except for a few outliers) but detection limit exceeded CP1.
 - Partial/ No Heat Treatment: all exceed CP1

Highlight: Full heat treatment is generally effective at reducing *E. coli*, fecal coliforms, and *Salmonella* in sampled digestate.

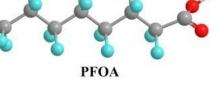


Per- and Poly-Fluoroalkyl Substances (PFAS)



- Per- and Poly-Fluoroalkyl Substances (PFAS) are referred to in media as "forever chemicals" due to their persistence in the environment.
- They are a concern as have been associated with numerous human health and ecotoxicological effects.
- PFAS have been used in many industrial and consumer products since the 1950s including fire-fighting foam, cookware, food packaging, carpets, and textiles.
- Regulatory actions have been taken in Canada and internationally to restrict use of some long-chain PFAS. Industry has shifted to using alternatives, including short-chain PFAS.
- A few US and Canadian jurisdictions have proposed or implemented regulatory requirements to address PFAS in sewage biosolids that are land-applied as fertilizers or soil amendments.
- A recent Canadian study (<u>Schwartz-Narbonne et al., 2023</u>) found high PFAS concentrations in fast food packaging, particularly in fiber-based products marketed as "compostable". PFAS are used in these products to improve mechanical strength and water resistance.
- See the appendix for a full tabular summary of the aggregated PFAS data

Heather Schwartz-Narbonne, Chunjie Xia, Anna Shalin, Heather D. Whitehead, Diwen Yang, Graham F. Peaslee, Zhanyun Wang, Yan Wu, Hui Peng, Arlene Blum, Marta Venier, and Miriam L. Diamond, *Environmental Science & Technology Letters* **2023** *10* (4), 343-349



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Paustenbach and Associates, 2021: <u>https://paustenbachandassociates.com/wp-content/uploads/2021/08/PFAS-PFOS.jpg</u> [Accessed 2024-03-11]

PFAS – Percent Detection

- Table shows % of samples with positive detection for each PFAS
- At least one PFAS detected in 86% of samples
- Long-chain PFAS subject to regulatory restrictions = fewer detections in digestate
 - Remaining detections possibly due to persistence of substances or breakdown of pre-cursors.
- Short-chain PFAS still in use = more detections in digestate
- Short-chain PFAS often partition to liquid phase = more detections in Separated Liquid vs. Separated Solid
- Little apparent difference between Agri-Food and SSO

Highlight: Short-chain PFAS more frequently detected reflecting more common use in products/ manufacturing due to regulations on long-chain PFAS.

Presence of PFAS in digestate (% of positive samples)

Analyte (Chain Length)		Ag	ri-Food (%)	SSO (%)			
		Whole (n = 12)	Sep. Liquid (n = 10)	Sep. Solid (n = 10)	Whole (n = 33)	Sep. Liquid (n = 7)	Sep. Solid (n = 6)	
L	PFBA (C4)	75%	100%	70%	76%	100%	0%	
hai	PFBS (C4)	0%	0%	0%	3%	14%	0%	
- -	PFPeA (C5)	17%	30%	0%	33%	29%	0%	
Short-Chain	PFHxA (C6)	17%	30%	20%	79%	86%	17%	
S	PFHpA (C7)	8%	20%	0%	3%	0%	0%	
	PFHxS (C6)	0%	0%	0%	0%	0%	0%	
	<u>PFOA (C8)</u>	50%	30%	10%	33%	14%	0%	
	<u>PFOS (C8)</u>	25%	0%	20%	6%	0%	0%	
2	PFOSA (C8)	33%	20%	0%	21%	43%	0%	
hai	PFNA (C9)	17%	0%	0%	0%	0%	0%	
Long-Chain	PFDA (C10)	0%	0%	0%	0%	0%	0%	
ů	PFDS (C10)	0%	0%	10%	0%	0%	0%	
	PFUnA (C11)	0%	0%	0%	0%	0%	0%	
	PFDoA (C12)	0%	0%	0%	0%	0%	0%	
	PFTrA (C13)	0%	0%	0%	0%	0%	0%	
	PFTeA (C14)	0%	0%	0%	0%	0%	0%	

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PFAS – PFOS and PFOA

Analyte	Agri-Food				SSO	SSO	CFIA Interim	
All values in ppb dry wt.	Whole (n = 12)	Separated Liquid (n = 10)	Separated Solids (n = 10)	Whole (n = 33)	Separated Liquid (n = 7)	Separated Solids (n = 6)	Compost: MECP 2019 (n = 1)	Standard: Biosolids
PFOS (C8)	1.7	<1.2	5.2	0.9	<1.1	<1.8	0.7	50
PFOA (C8)	0.6	0.7	0.9	0.8	0.4	<0.9	1.3	-

PFAS in digestates (95th Percentiles) compared to Compost and CFIA interim standard

 Perfluorooctane Sulfonate (PFOS) and Perfluorooctanoic Acid (PFOA) are most studied, and regulated PFAS (e.g., Canadian EPA toxic; Stockholm Convention designations)

- Proposed Regulations under development for sewage biosolids and other related fertilizing residuals:
 - **Canada:** PFOS 50 ppb, interim standard proposed by CFIA
 - **Quebec:** TBD Standards under development by provincial environment ministry

Highlights: PFOS and PFOA content in sampled digestate are mostly below detection limit, and all PFOS results are well below the threshold proposed for sewage biosolids by CFIA.



🔲 Agri-Food Digestate 📃 SSO Digestate 📃 SSO Compost

Plastics and Foreign Matter (FM)

Comparison Between Agri-Food and SSO Digestate

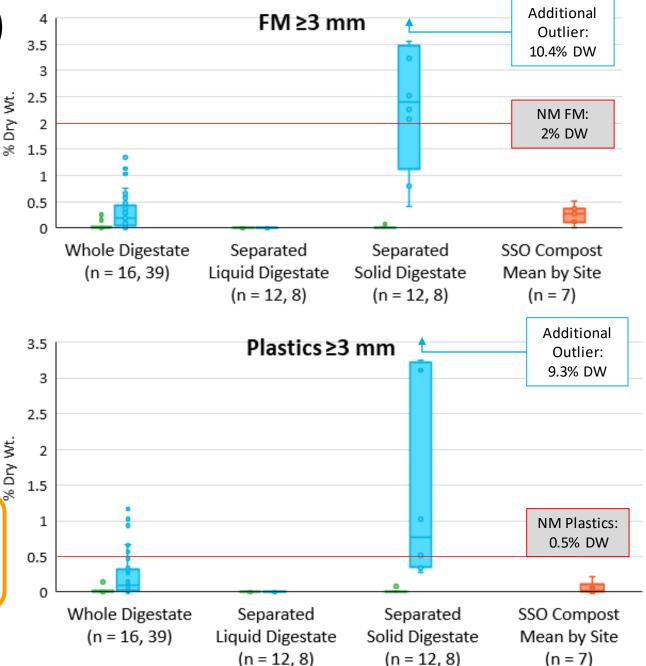
- Higher plastics and foreign matter (FM) levels in digestate associated with facilities that receive SSO feedstocks – operators report 12 – 20% contamination on a wet basis in raw municipal SSO
- Practically zero plastics and FM in agri-food digestates

 likely due to low-contaminant feedstocks
- Practically zero plastics and FM in separated liquid digestates – separation acts like screening step and concentrates contaminants in separated solid

Comparison Between Compost and Digestate

 Plastics and FM in compost lower than separated solid digestate – see concentrating effect when digestate is dewatered

Highlight: Plastics and FM higher in sampled SSO digestates due to higher contamination in feedstocks which concentrate into separated solids.



AD Study – Preliminary Highlights

- Digestate is a good source of nutrients (N-P-K), and nutrient profile of sampled digestates are similar despite different feedstocks (see data in Appendix).
- Metals content are mostly well below CM1 with a few exceptions:
 - **Cu/ Zn:** elevated levels in sampled agri-food digestates likely due to nutritional additives used in livestock feed found in manure feedstocks. Values still below Category A compost limits.
- Full heat treatment is generally effective at reducing *E. coli*, fecal coliforms, and *Salmonella*.
- Short-chain **PFAS** more frequently detected, reflecting more common use in products/ manufacturing due to regulations on long-chain PFAS.
 - **PFOS** and **PFOA** content mostly below detection limit, and all well below PFOS threshold proposed for sewage biosolids by CFIA.
- Plastics and Foreign Matter higher in sampled SSO digestates and most concentrated in separated solids

 likely due to higher contamination in municipal SSO feedstocks received.



Acknowledgements

THANK YOU FOR YOUR PARTICIPATION & SUPPORT!

- Participating facilities
 - Farmers
 - Anaerobic Digester Owners and Operators
- MECP Laboratory Services Branch
- E3 Laboratories Inc.
- University of Guelph:
 - School of Engineering
 - Arkell Research Station
- Agriculture and Agri-Food Canada London
- MECP and MAFA staff and students
- Contact us at:
 - <u>Cecily.Flemming@Ontario.ca</u>
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Biogas Safety Awareness Online Course – UPDATED 2024!

Offered through University of Guelph Ridgetown Training

https://bdc.ridgetownc.com/takecourse/energy-courses/biogas-safety-online-course/

- Explains common hazards at agricultural biogas system.
- Describes best actions to take to reduce the risk of unsafe incidents.
- Takes ~ two hours for completion. Includes quizzes and a final test.
- \$40 registration

Suitable for:

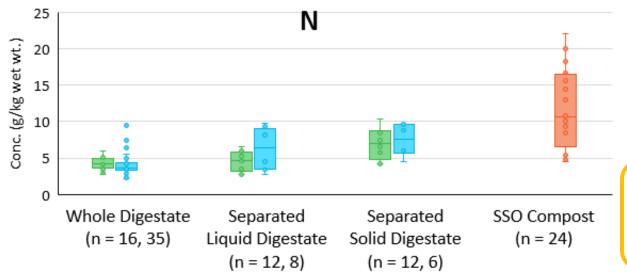
- Staff and owner/operators
- Contractors who perform regular service at digesters
- Farm family



Appendix – Providing more Data Detail

- Primary Nutrients (N-P-K) Box Plots
- Total and Volatile Solids Box Plots
- Regulated Metals
 - Cobalt and Molybdenum Box plots
 - Selenium and Nickel Box plots
 - Full Summary of metals (95th Percentiles)
- PFAS Full Summary (95th Percentiles)

Primary Nutrients, Total (N-P-K)



📕 Agri-Food Digestate 📕 SSO Digestate 📕 SSO Compost

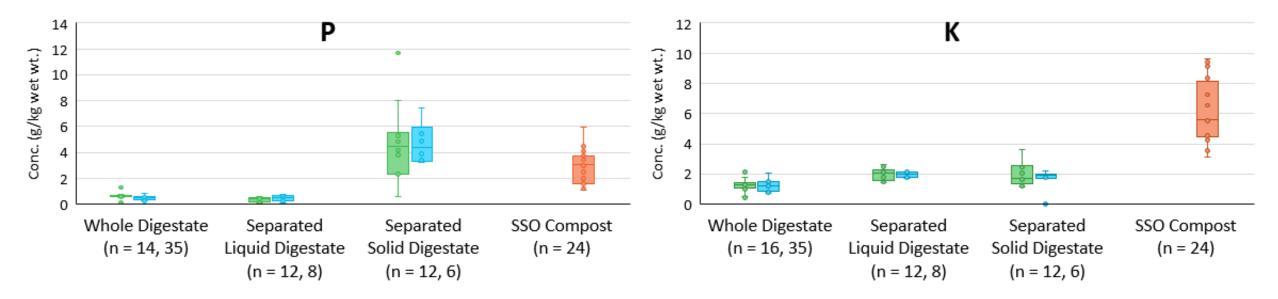
Comparison Between Agri-Food and SSO Digestate

- Agri-food and SSO digestates similar in nutrient profile
- Higher solids materials = higher nutrients (see appendix for summary of total/ volatile solids)
- As expected, P partitions to the separated solids

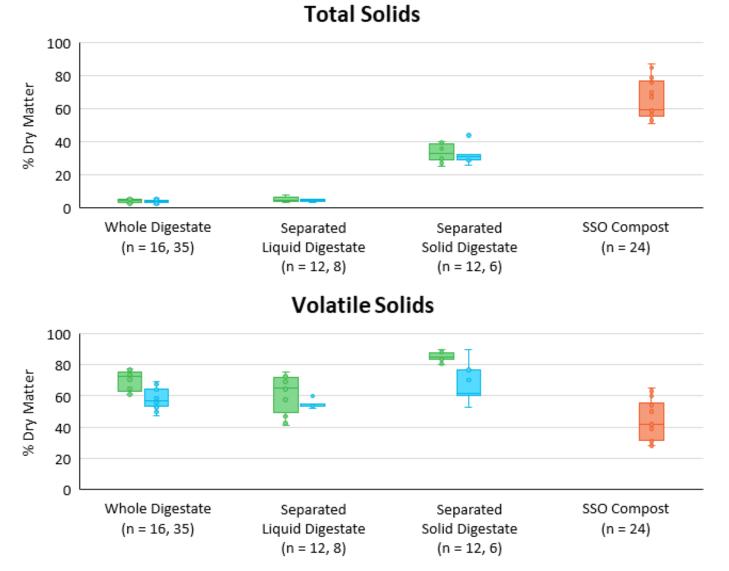
Comparison Between Compost and Digestate

- Compost generally higher in nutrients than digestate when compared on a wet weight basis – compost higher in solids
- Exception is phosphorus in separated solid digestate

Highlights: Digestate is a good source of nutrients, and nutrient profile of sampled digestates are similar despite different feedstocks.



Total and Volatile Solids



Agri-Food Digestate SSO Digestate SSO Compost

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Regulated Metals (Co, Mo)

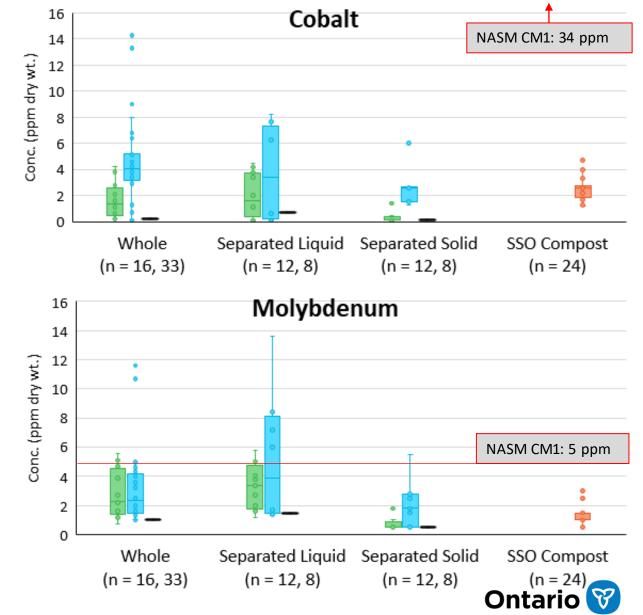
• Co, Mo are trace elements with benefits to AD

Comparison Between Agri-Food and SSO Digestate

- All results for Co below NASM CM1 standard.
- Most results for Mo below CM1 standard.
- Co in SSO digestate generally higher than agrifood, likely due to more Co supplements used.

Comparison Between Compost and Digestate

- Co and Mo:
 - Digestate comparable to SSO compost in terms of central tendency (median).
 - Digestate more variable possibly due to smaller dataset, or use of trace element supplements containing Co and Mo.



Regulated Metals (Se, Ni)

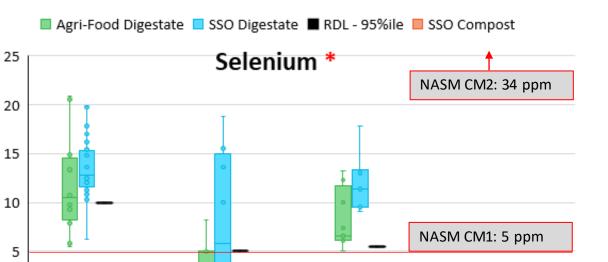
• Se and Ni are trace elements with benefits to AD.

Comparison Between Agri-Food and SSO Digestate

- Most results for Se exceed the CM1 standard, but are below the CM2 standard.
- Se in SSO digestate generally higher than agri-food.
- All results for Ni are below the NASM CM1 standard.

Comparison Between Compost and Digestate

- Se: Digestate notably higher than SSO compost.
- Ni: Digestate comparable to SSO compost.



Separated Liquid

(n = 12, 8)

Separated Liquid

(n = 12, 8)

Nickel

Separated Solid

(n = 12, 8)

Separated Solid

(n = 12, 8)

SSO Compost

(n = 24)

SSO Compost

(n = 24)

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NASM CM1: 62 ppm

Conc. (ppm dry wt.)

0

35

30

25

20

15

10

5

0

Conc. (ppm dry wt.)

Whole

(n = 16, 33)

Whole

(n = 16, 33)

*Se in digestate being re-tested due to potential false positives in initial analysis.

Regulated Metals – Full Summary

Regulated Metals (95th Percentiles) compared to Compost and NM Standards

Analyte All values in ppm dry weight	Agri-Food Digestate: Whole (n = 16)	Agri-Food Digestate: Separated Liquid (n= 12)	Agri-Food Digestate: Separated Solids (n = 12)	SSO Digestate: Whole (n = 33)	SSO Digestate: Separated Liquid (n = 8)	SSO Digestate: Separated Solids (n = 7)	SSO Compost: MECP 2011 Study (n = 24)	CM1	CM2
Arsenic	<7.7	<5	<8.3	<10	<5	<5	8.7	13	170
Cadmium	0.5	<0.4	<0.2	<0.2	0.8	<0.1	0.6	3	34
Chromium	40.9	32.9	26	15.5	12	15.6	26.6	210	2,800
Cobalt	4.1	4.3	1.1	14.1	6.6	5.7	4	34	340
Copper	279.6	223.6	71.2	70.9	46.2	45.8	69.4	100	1,700
Lead	14.1	7.7	5.7	18.9	20.5	47.4	67.7	150	1,100
Mercury	0.1	<0.3	<0.02	0.1	<0.2	0.06	0.1	1	11
Molybdenum	5.3	5.6	1.5	4.5	6.3	2.8	2.9	5	94
Nickel	32.3	17.5	16.4	13.7	13.5	11.8	13.7	62	420
Selenium	14.1	7.2	12.9	19.4	18.3	11.2	0.3	2	34
Zinc	388.8	649.5	241	269.4	261.8	287.4	190	500	4,200



PFAS – Full Summary

PFAS in digestates (95th Percentiles) compared to Compost

Analyte All values in ppb dry wt.	Agri-Food Digestate: Whole (n = 12)	Agri-Food Digestate: Separated Liq. (n = 10)	Agri-Food Digestate: Separated Solid (n = 10)	SSO Digestate: Whole (n = 33)	SSO Digestate: Separated Liq. (n = 7)	SSO Digestate: Separated Solid (n = 6)	SSO Compost: MECP 2019 (n = 1)
PFBA (C4)	8.8	8.5	2.7	6.9	11.4	<1.8	1.7
PFBS (C4)	<1.2	<1.2	<1.7	1.1	2	<1.8	6
PFPeA (C5)	3	2.6	<1.7	5.1	1.7	<1.8	1.3
PFHxA (C6)	1.4	1.3	2.9	3.1	3.2	<1.8	6.6
PFHpA (C7)	<1.2	<1.2	<1.7	1.2	<1.1	<1.8	0.34
PFHxS (C6)	<2.5	<2.4	<3.4	<2.2	<2.2	<3.6	<0.2
PFOA (C8)	0.8	0.8	1.1	0.8	<0.5	<0.9	1.3
PFOS (C8)	1.7	<1.2	5.2	1.3	<1.1	<1.8	0.71
PFOSA (C8)	3.1	1.4	<0.8	1.6	1.1	<0.9	0.09
PFNA (C9)	0.9	<0.6	<0.8	<0.5	<0.5	<0.9	0.23
PFDA (C10)	<0.6	<0.6	<0.8	<0.5	<0.5	<0.9	0.45
PFDS (C10)	<1.2	<1.2	1.9	<1.1	<1.1	<1.8	<0.1
PFUnA (C11)	<0.6	<0.6	<0.8	<0.5	<0.5	<0.9	0.1
PFDoA (C12)	<1.2	<1.2	<1.7	<1.1	<1.1	<1.8	0.17
PFTrA (C13)	<1.2	<1.2	<1.7	<1.1	<1.1	<1.8	<0.1
PFTeA (C14)	<1.2	<1.2	<1.7	<1.1	<1.1	<1.8	0.12