A Novel Application of Biopile Technology : Addressing Phthalate Ester Contamination

Remediation of a former chemical plant

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Phthalate Contamination: Remediation Options and Potential of Biotreatment



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What are phthalates?

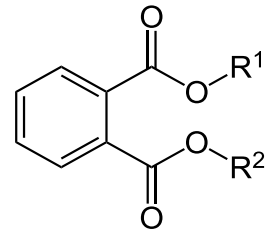
Phthalates (PAEs) are synthetic compounds widely used as additives in plastic production. They are:

- **Present in various daily products** including food packaging, toys and personal care product;
- **Mobile and persistent**, they have been globally detected in the environment
- **Toxic** (including endocrine disruption effects and reproductive toxicity for humans)

Contaminants of significant environmental concern







Generic structure of phthalates molecules

Phthalate management practices in Quebec

In Québec, regulatory cleanup criteria are specific for individual phthalate compound.

DEHP is the most frequently detected phthalate in soils due to its widespread use in plastic products[3] and its characteristics making it persistent in the environment.

Parameters	Unit	Remediation criteria		
		A	B / Residential	C / Industrial-commercial
Di(2-ethylhexyl) phthalate	mg/kg	--	--	60
Dimethyl phthalate	mg/kg	--	--	60
Diethyl phthalate	mg/kg	--	--	60
Di-n-butyl phthalate	mg/kg	0.2	6	70,000
Benzyl butyl phthalate	mg/kg	--	--	60
Di-n-octyl phthalate	mg/kg	--	--	60

There is **no remediation technology** approved by the Quebec Ministry of Environment **for the remediation of phthalate impacted soils**



Currently, soils contaminated with phthalates are **excavated** and **sent for disposal at a secured landfill**.

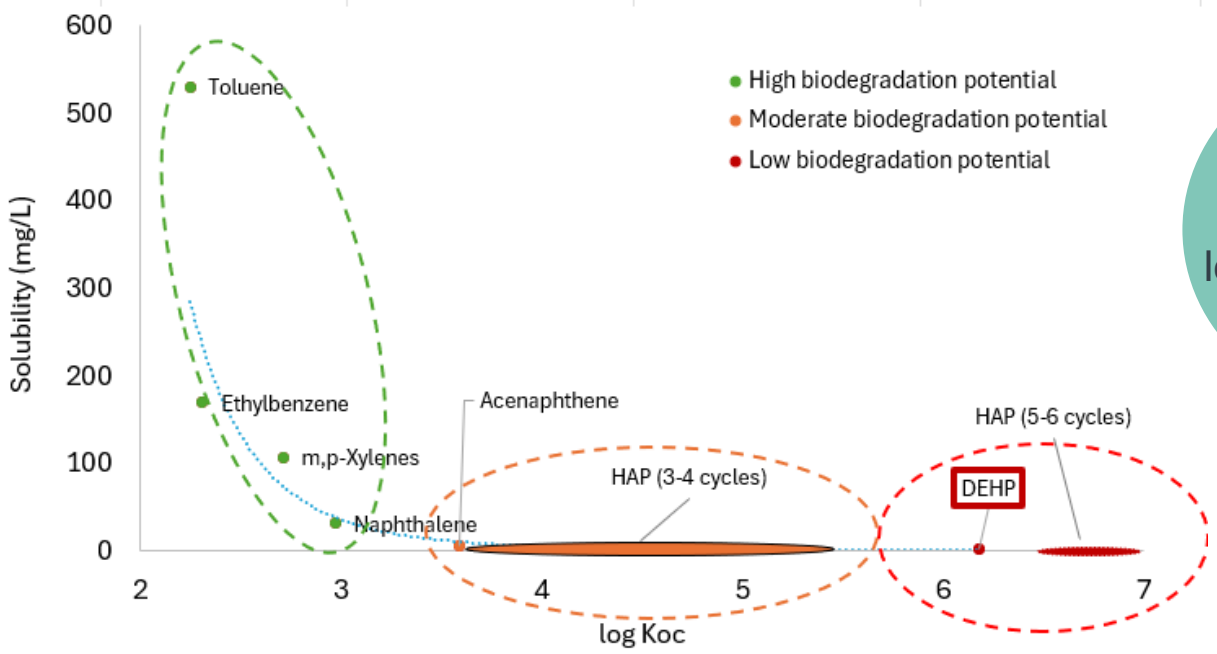
Existing remediation technologies

Technology	Maturity	Reference
Thermal desorption	Pilot test	Ortec, France[1]
Bioremediation	Laboratory - pilot	Chen et al., 2022[2]
Adsorption and partial immobilization using biochar	Laboratory	Tran et al., 2022[3]
Chemical oxidation (e.g. hydrogen peroxide)	Laboratory	Ordoñez-Frías et al., 2020[4]

Applicability of biotreatment to phthalates

DEHP chemical characteristics

1. **Vapor pressure**
   ~10^{-7} mmHg or 10^{-5} Pa (25 °C) → **low volatility**
2. **Solubility in water**
   3,0 x 10^{-3} mg/L (20 °C) → **low solubility in water**
3. **Organic carbon/water partition coefficient**
   Log Koc 5.41 to 6.18 → **high affinity with organic carbon in soil**



Based on its caracteristics, DEHP presents a low biodegradation potential.

Applicability of biotreatment to phthalates

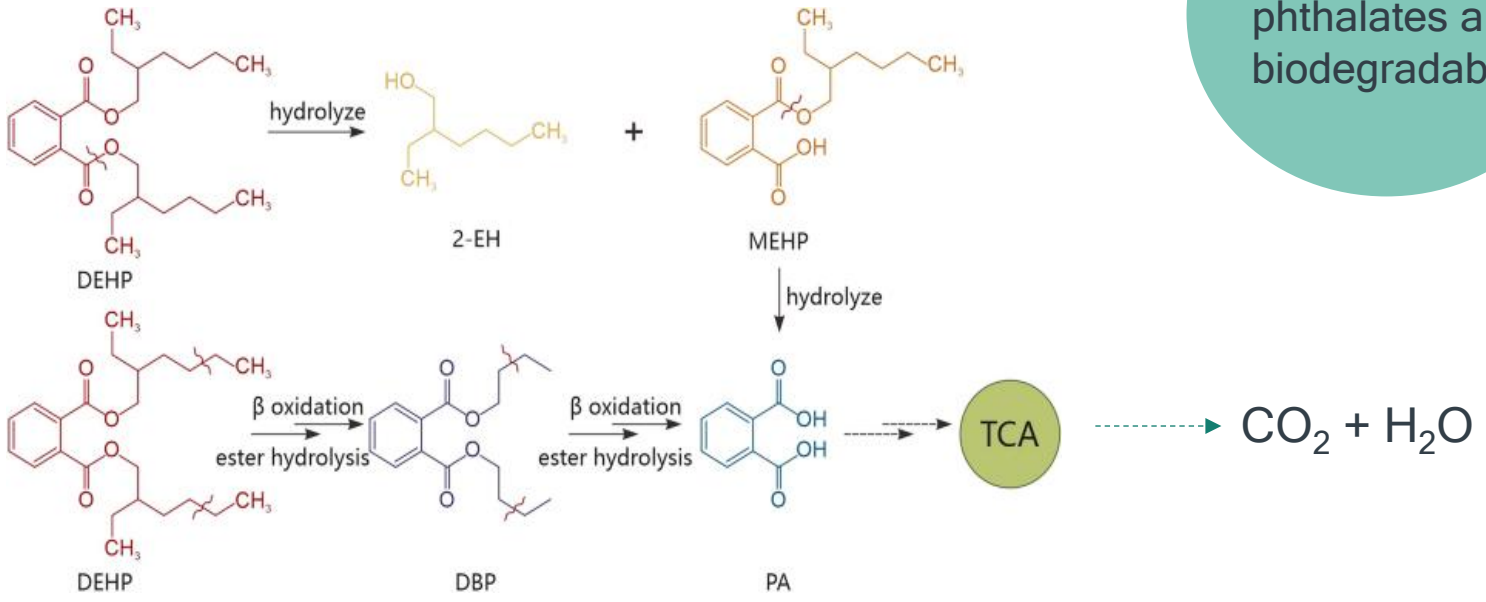
Biodegradability

Specifically, for **DEHP**:

- 75% biodegradation in 42 days in sandy soil mixed with gravel/till taken from a plasticizer manufacturing site[5]
- Half-life from 2 to 77 days in different type of soil[6]
- Half-life of 20 days in silty soil[7]

Different metabolic degradation pathways

Some studies show that phthalates are biodegradable

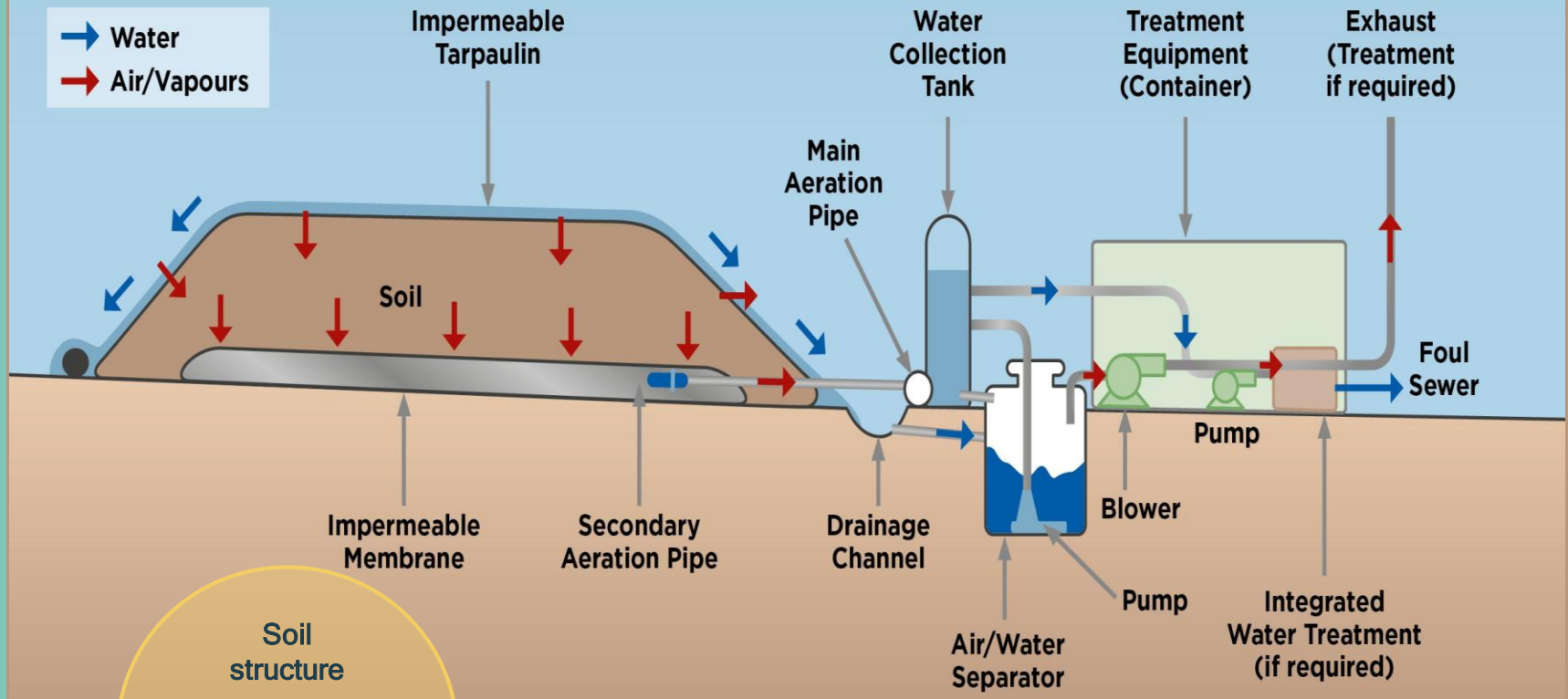


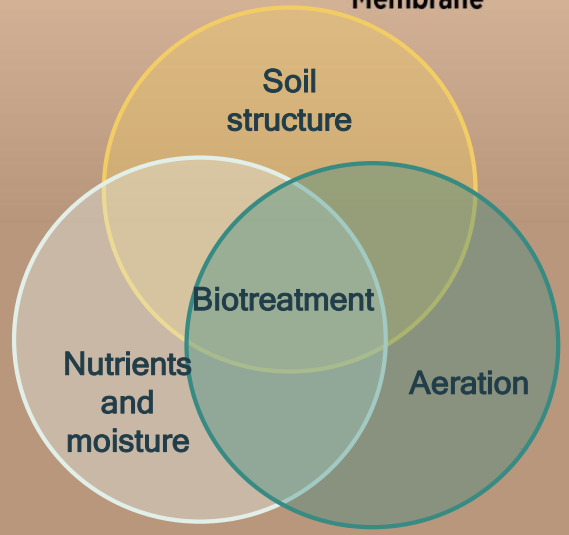
Proposed pathway of DEHP degradation by a specific strain in electronic waste soil (Chang et al., 2022[8]). DEHP : di(2-éthylhexyl) phtalate; 2-EH : 2-Ethylhexanol; MEHP : Monoethylhexyl phthalic acid; DBP : Dibutyl phthalate; PA : phthalic acid; TCA : tricarboxylic acid cycle.

What is biotreatment?

1. Contaminated soils are excavated and placed on an **impermeable barrier**. Organic amendments are added to improve soil structure.
2. A piping network insure proper air circulation and the collection of process air and water.
3. **Soil is mixed** frequently during treatment for amendment addition and to **increase contaminant availability**.
4. **Key parameters** are **monitored** and adjusted (oxygen, nutrients, moisture etc.) to **promote biodegradation** by soil microorganisms.
5. Contaminant and **volatilized** and/or biologically **degraded.**

Treatment Process: Ex Situ Biopile





What is biotreatment?









Laboratory testing and pilot test



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Laboratory testing – Microbial characterization

The measurement of total and specific bacterial count provides an **indication of the soil's biological activity** and its **capacity to biodegrade contaminants**. These results help selecting the **amendment** to optimize bioremediation performance.

1 **Total bacterial count**

~Overall population of microorganisms present in the soil (aerobic conditions).

2 **Specific bacterial count**

Microorganisms capable of growing in the using solely a specific contaminant as a **source of carbon or energy**. This allows for the quantification of microbial populations likely to contribute to the biodegradation of contaminants.



Petri dish

Laboratory testing - Amendment selection









Amendment	Total bacterial count UFC/g d.b.	DEHP specific bacterial count UFC/g d.b.	Total/ specific	Used for
Horse litter	1,5,E+09	1,5,E+08	10%	Nutrients source Soil structuration
Chicken manure	3,5,E+07	4,1,E+04	10%	Nutrients source
Peat moss	9,5,E+04	2,0,E+03	2%	Acidification Moisture retention
Alfalfa/lucerne	2,3,E+04	8,6,E+03	37%	Nutrients source Heat generation

All amendments contain DEHP degrading bacteria
Horse litter presents the **highest** total and DEHP specific **bacteria counts**

Alfalfa has the **greatest proportion of DEHP bacteria**

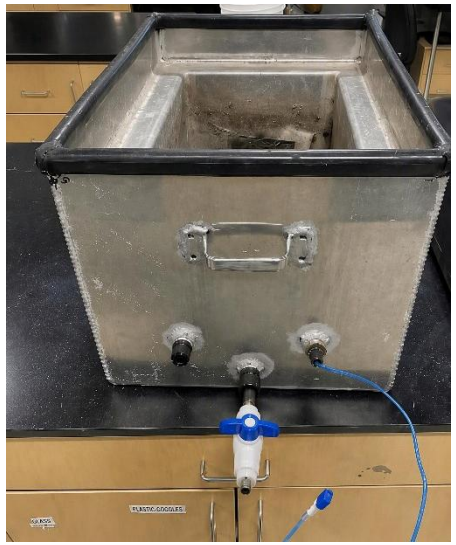
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Treatability study

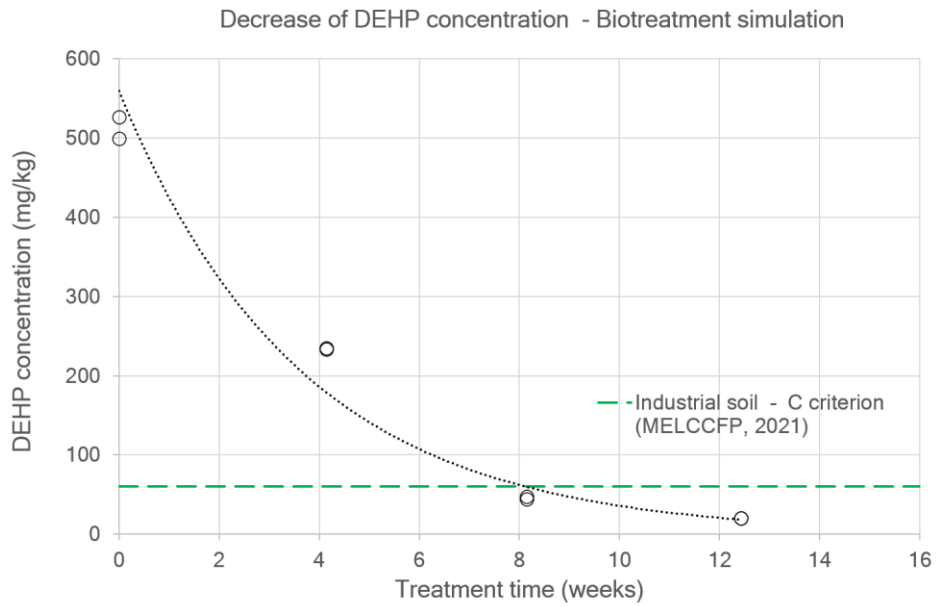
Small scale simulation of the biotreatment process.

1. Simulation conducted on 20 kg of soil place in a **mesocosm.**
2. **Hors litter** used to **improve soil structure.**
3. **Aeration** and adjustment of **moisture** and **nutriments** levels.









96% reduction of DEHP concentrations after **12 weeks**

Strong potential of biotreatment to address **DEHP contamination**

* The “RESC” criterion in the Regulation on the Landfilling of Contaminated Soil is also 60 mg/kg for DEHP

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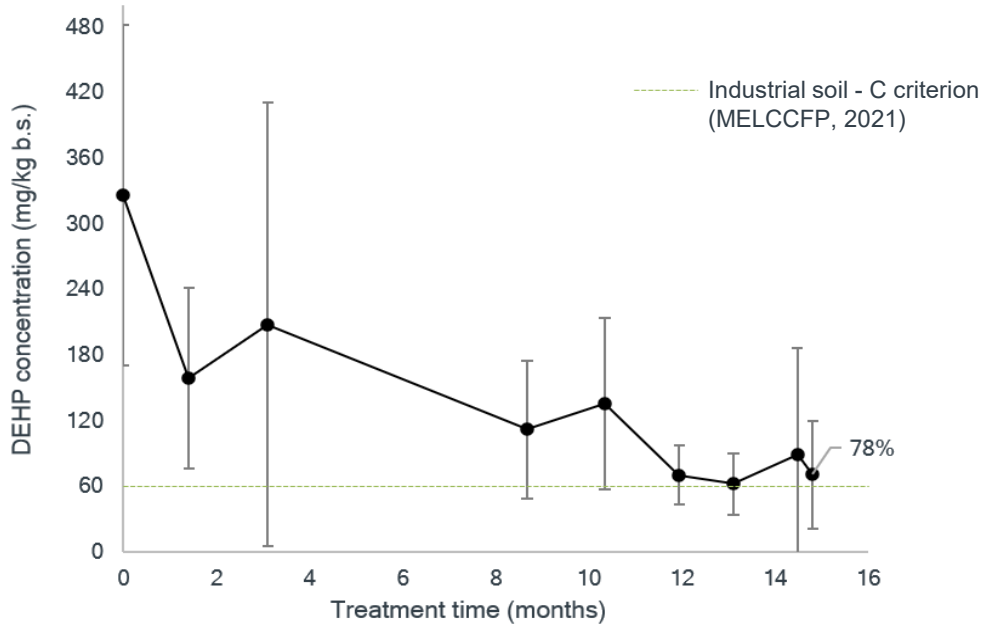
Pilot test

1. **Biopile of 2,360 metric tons** of soil contaminated with phthalates from an industrial site.

2. Addition of **fertilizer** and **horse litter** (less than 10% v/v).



Soil treatment facility located in Montreal East, formerly owned by Englobe, now owned by Biogenie (Ortec)



78 % of reduction of DEHP concentrations after 15 months

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Pilot test – Challenges and optimization

- **High pH** conditions during the pilot test, ranging from 8,4 to 9,9 – may have delayed/decreased biodegradation.

- Treatment initiated in late Fall with **clayey soil** resulted in high moisture and poor soil structure thus extending treatment time.



DEHP can biodegrade at full scale, but **maintaining biological treatment parameters is critical** to avoid extending the duration of treatment

Full-scale treatment



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Site history and project scope

Industrial production (1932-2008)

Resins, plasticizers, polymers, wood stains, herbicides, plastics (including polystyrene) and synthetic fibres.

Mixed contamination

- Petroleum hydrocarbons / PAHs / VOC / Asbestos
- Metals, including mercury
- **Phthalates**: plasticizers that soften plastics and increase flexibility

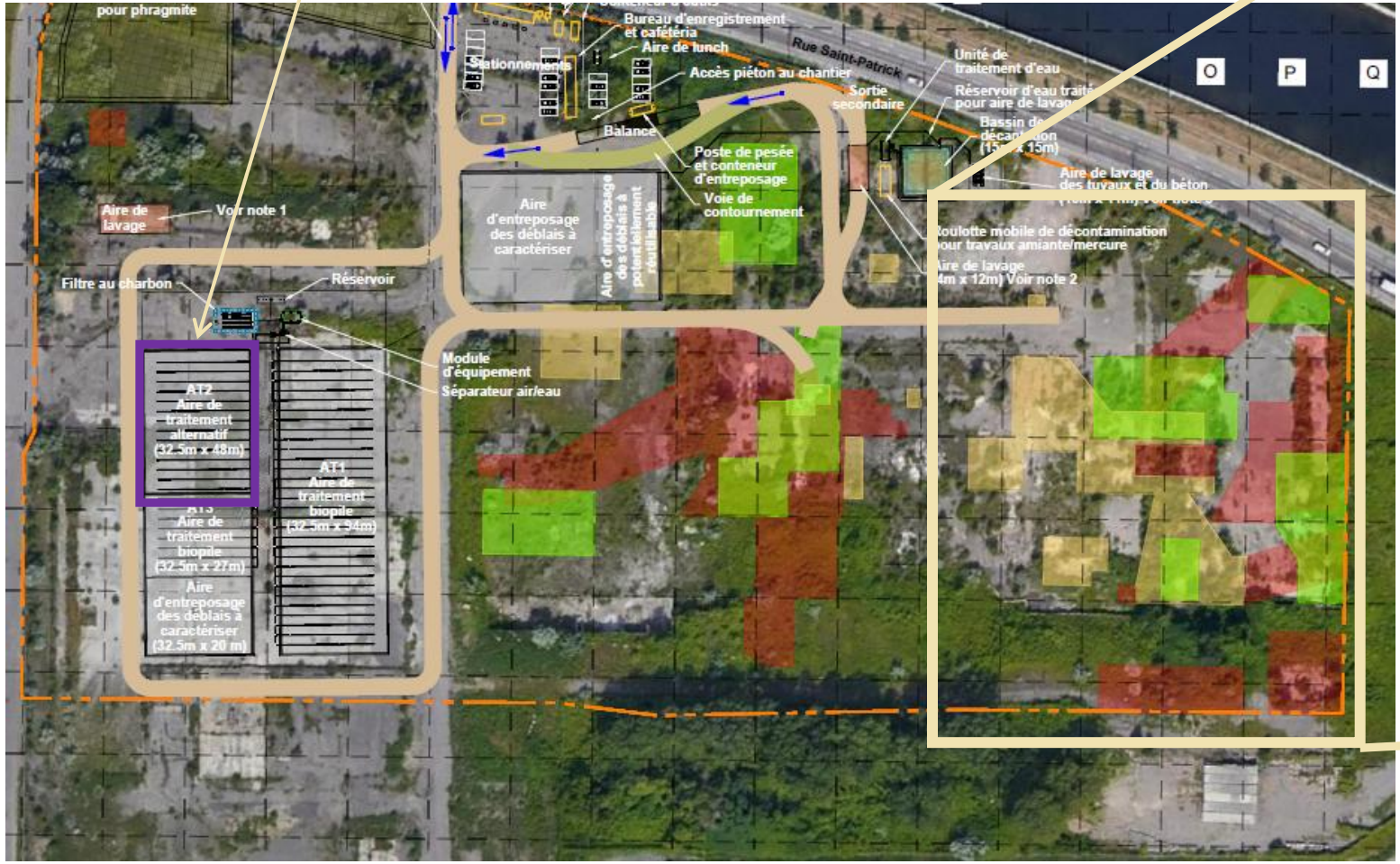
Remediation scope

Activity	Planned	Actual
Total excavation	148,750 t	314,500 t
On-site soil treatment	32,960 t	32,000 t
Phthalate thermal treatment	7,350 t	-
Phthalate biotreatment	-	**9,191 t**

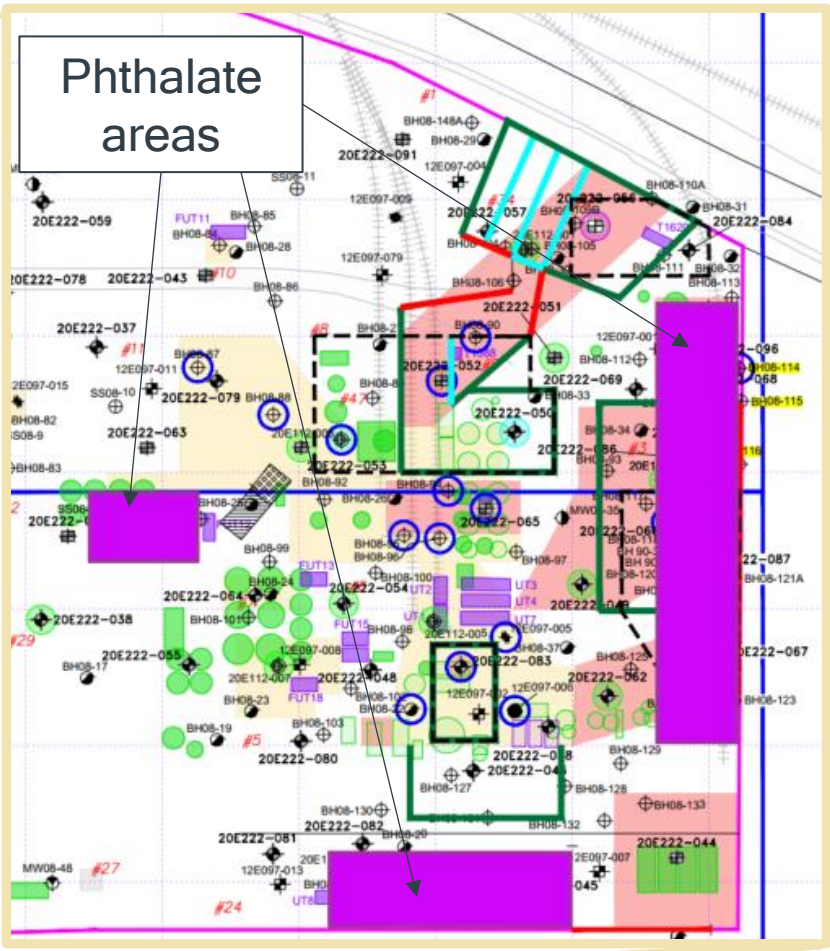


Contamination distribution vs. other contaminants

Phthalate area on the treatment pad



Phthalate areas



Phthalate biotreatment

Treatment approach

Phthalate biotreatment replaces the thermal treatment specified in the contract, significantly reducing costs.



Soil segregation

Recalcitrant soils are kept separate from soils contaminated with petroleum hydrocarbons and VOCs



Amendment selection

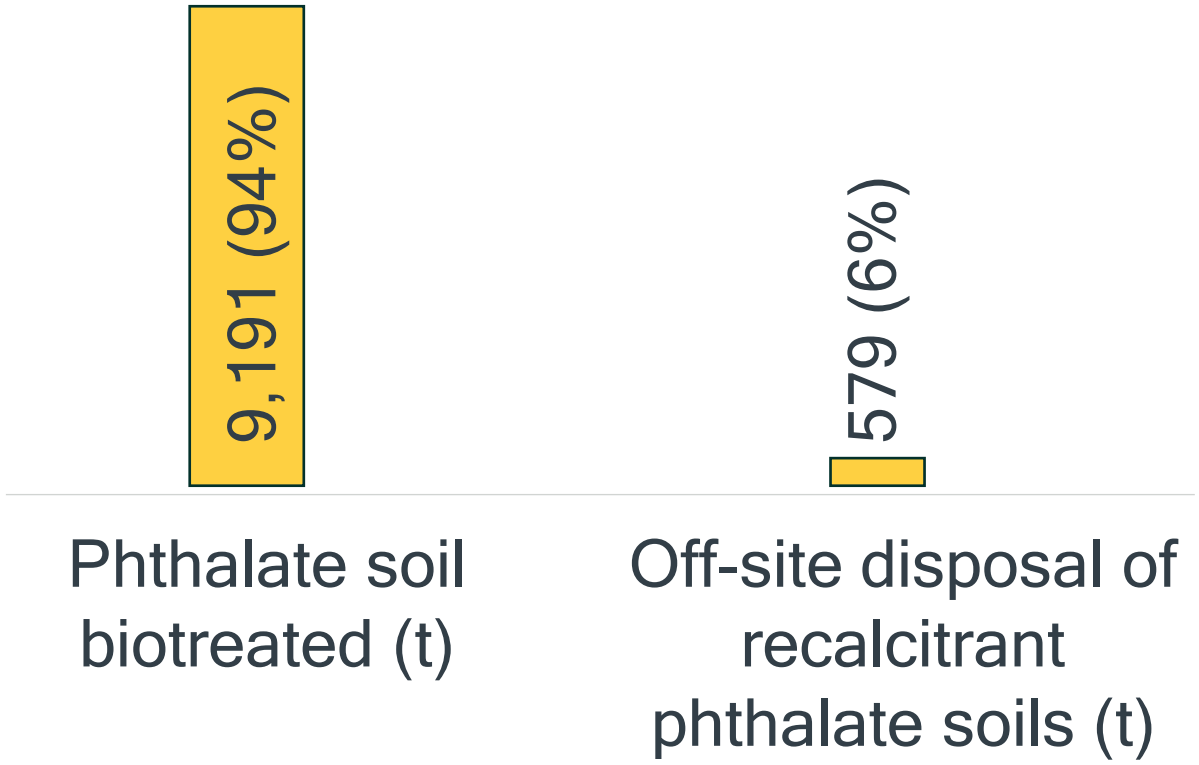
Organic: Wood chips from tree cutting improve clay soil structure and aeration. Alfalfa promotes contaminant biodegradation.

Inorganic: Nitrogen and phosphorus promote contaminant biodegradation.



Final results: Biotreated soil vs. off-site disposal

Summary of quantities







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Challenges

- **Clay soil** requiring adequate amendments
- **Longer treatment time** than for other contaminants (PHCs, PAHs, VOCs)
- **Variability in results** during treatment
- **Monitoring of biotreatment** parameters to optimize treatment

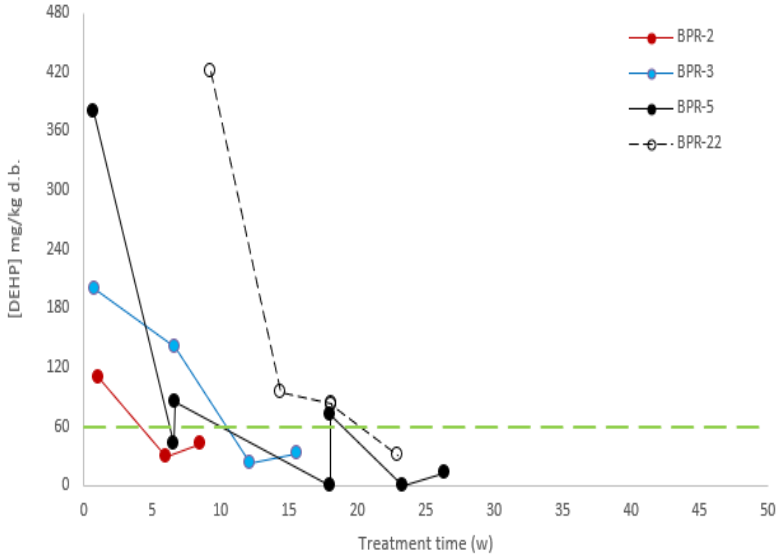


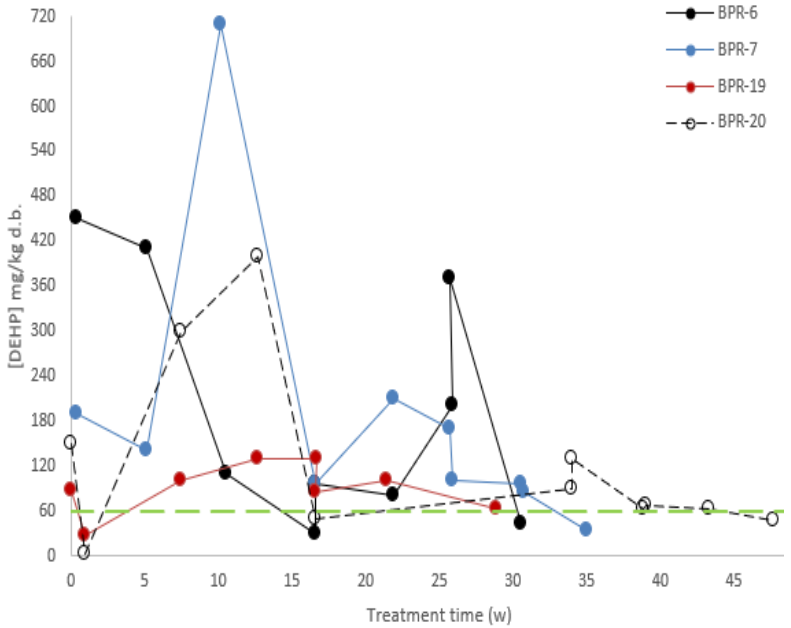


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Monitoring, treatment performance and variability in results

Contaminant degradation curves





Monitoring of biotreatment parameters

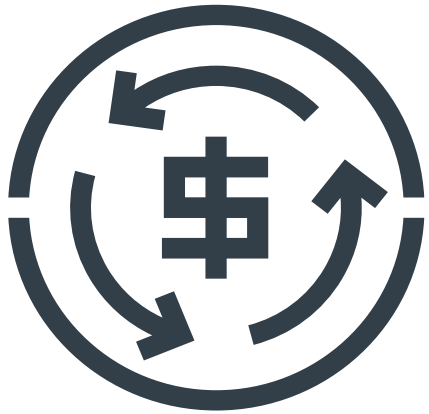
- Ammoniacal nitrogen
- Nitrate and nitrite
- Available phosphorus
- Bacterial counts
- pH and temperature

Treatment performance

- Concentrations decreased by **70% to 95%** after **7 to 20 weeks** of treatment
- Up to **35 weeks** of treatment for one soil batch that did not meet the reuse criteria
- Temperature increased by **10 °C** in soils amended with alfalfa

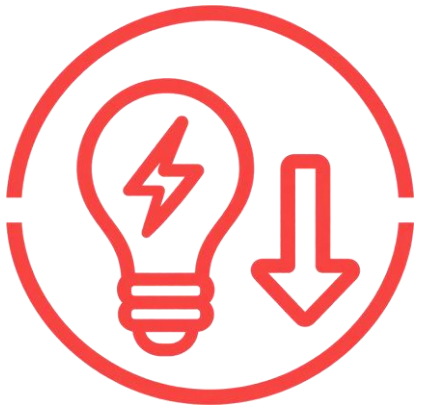
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Cost savings from biotreatment



$1,400,000

saved **by biotreating 9,191 tonnes** of soil compared with thermal treatment and more than **$370,000** compared with off-site disposal.



8 GWh

Energy saved compared with thermal treatment

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Full-scale results and lessons

Soil segregation: We treated soils in a dedicated zone of the treatment pad for closer monitoring and potentially longer treatment.

Biotreatment approach: Biotreatment in place of the planned thermal treatment. Alfalfa amendment enhanced biodegradation.

Outcome: We biotreated 94% of phthalate soils. Only 6% required off-site disposal.

Challenges: Clay-rich soils and variable analytical results required representative sampling and **a detailed treatment monitoring**.

Performance: Concentrations fell by up to 95% after 20 weeks. One batch needed 35 weeks. Alfalfa raised soil temperature by **10 °C**.

Treatment timing: Starting treatment in warm weather would have reduced heat losses and preserved the effectiveness of the amendments.

Summary



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Presentation wrap-up

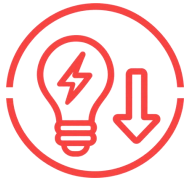
1. **Background.**
   Phthalates served as plasticizers to soften plastics and are widely detected in the environment

2. **Recalcitrant**
   Phthalates such DEHP is known as a recalcitrant compound.

3. **Potential**
   Laboratory and pilot tests results showed great potential of biological treatment by biopiles for soil contaminated by DEHP.

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Benefits of phthalate biotreatment



Energy saved

8 GWh of **energy saved** compared with thermal treatment.

Documented cost savings

Biotreating 9,191 t of soil saved $370,000 compared with off-site disposal and more **than $1.4 million** compared with thermal treatment.



Alternative to thermal treatment

Biotreatment allows the treatment of **94%** of the phthalate-contaminated soils.

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Q&A session



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