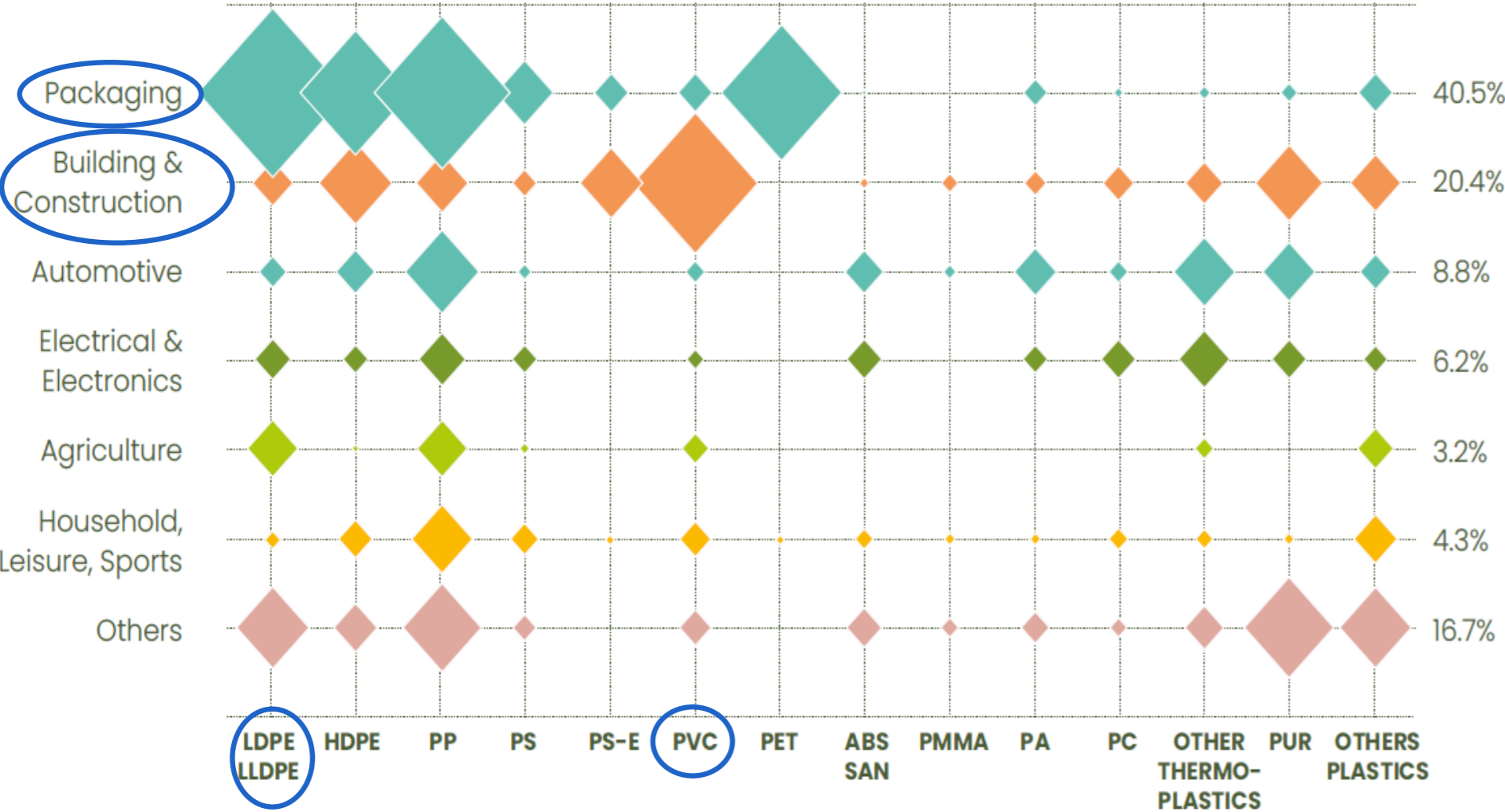


# SOLVENT-BASED RECYCLING TOWARDS RECOVERY OF TiO<sub>2</sub> FROM WASTE PLASTICS

CORFU 2022, 17<sup>th</sup> June

Elisabetta Carrieri, Steven De Meester

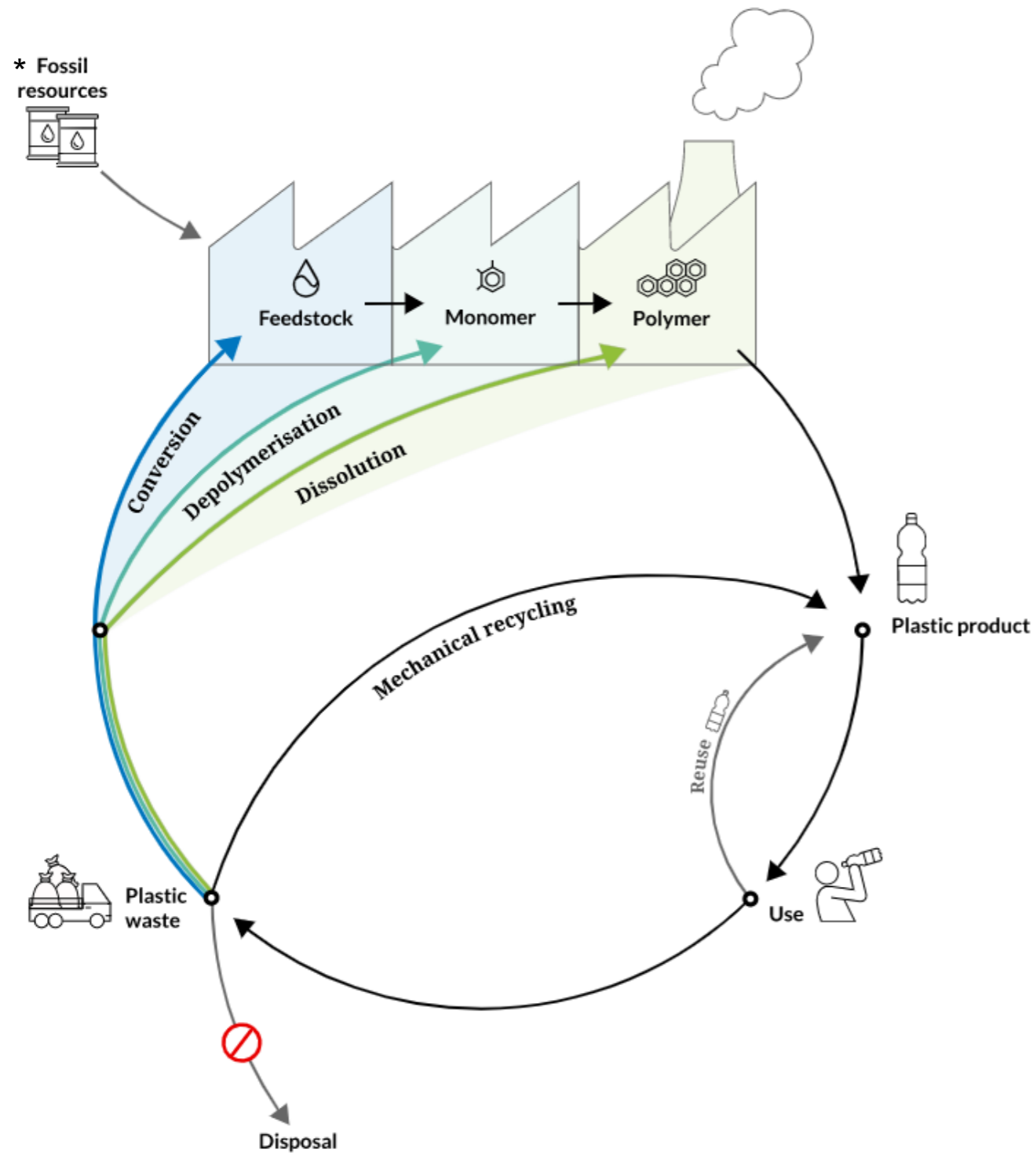
# PLASTICS (WASTE) PRODUCTION



**PLASTICS DEMAND IN EU**  
49.1 Mt

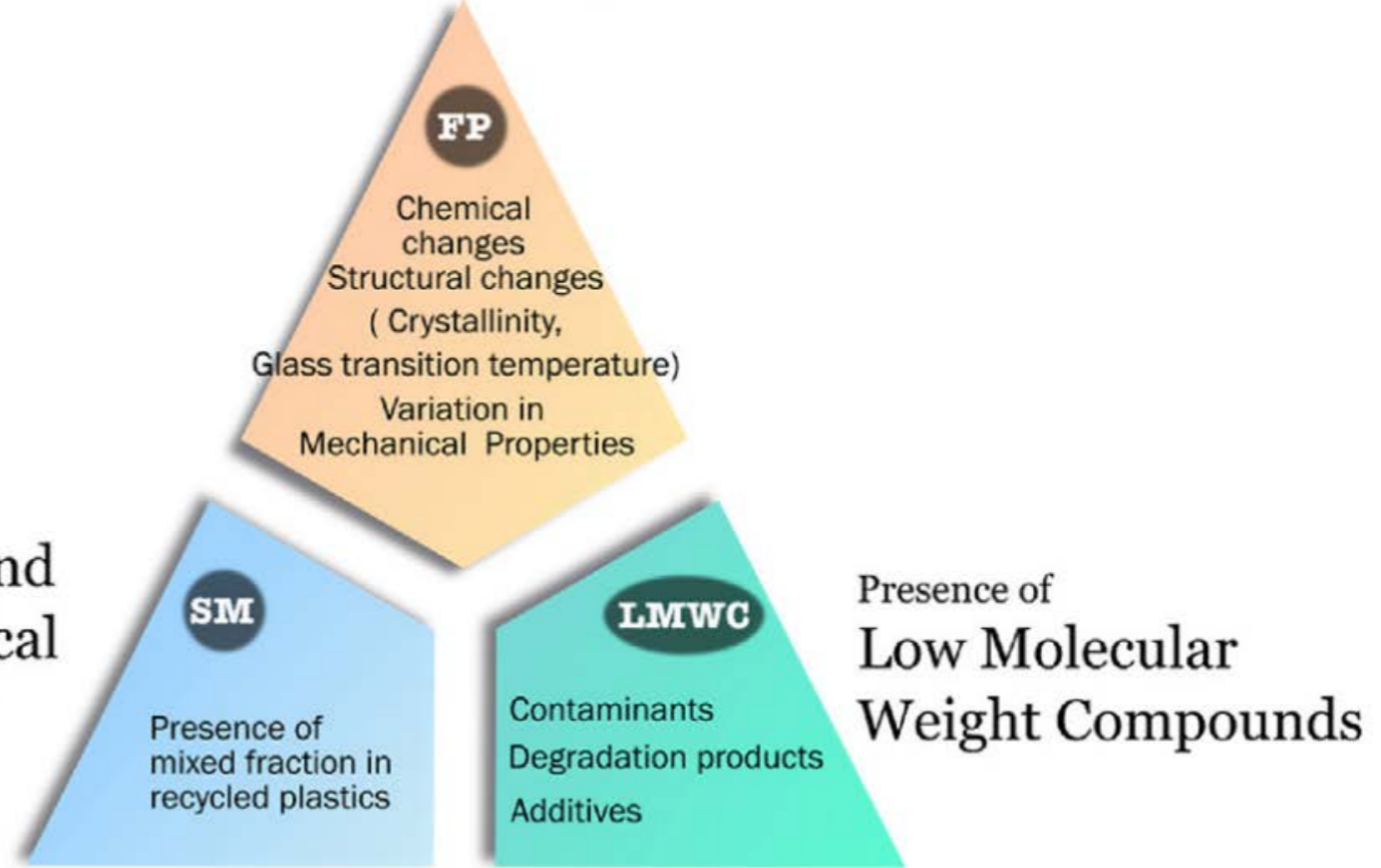


# PLASTICS RECYCLING ROUTES



Structural and Morphological Assessment (Composition)

Feasibility of production \*\* and Stability



\*Figure from <https://cefic.org/a-solution-provider-for-sustainability/chemical-recycling-making-plastics-circular/>

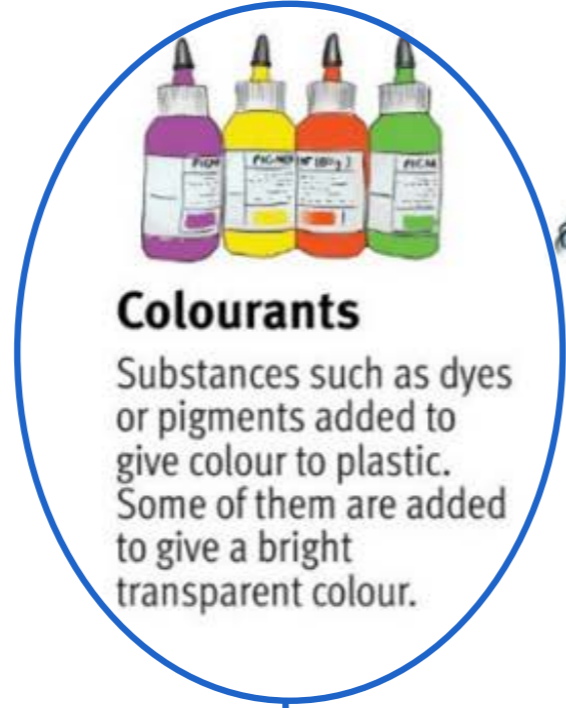
\*\*Figure from Cruz Sanchez (2020) Plastic recycling in additive manufacturing: A systematic literature review and opportunities for the circular economy.

# MANY ADDITIVES: WHY $TiO_2$ ?



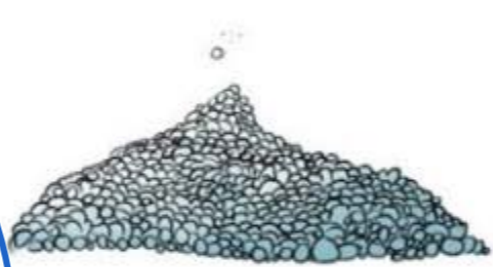
**Functional**

Includes, for example, stabilizers, antistatic agents, flame retardants, plasticizers, lubricants, slip agents, curing agents.



**Colourants**

Substances such as dyes or pigments added to give colour to plastic. Some of them are added to give a bright transparent colour.



**Fillers**

Added to change and improve physical properties of plastics. They can be minerals, metals, ceramics, bio-based, gases, liquids, or even other polymers.



**Reinforcement**

Used to reinforce or improve tensile strength, flexural strength and stiffness of the material. For example: glass fibres, carbon fibres.

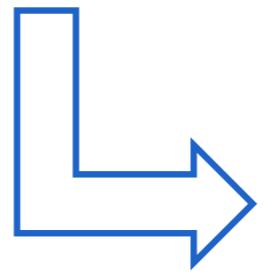


**NIAS**

Non-intentionally added substances. They arrive in products from processes such as reaction by-products or breakdown products.

Dyes  
(14%)

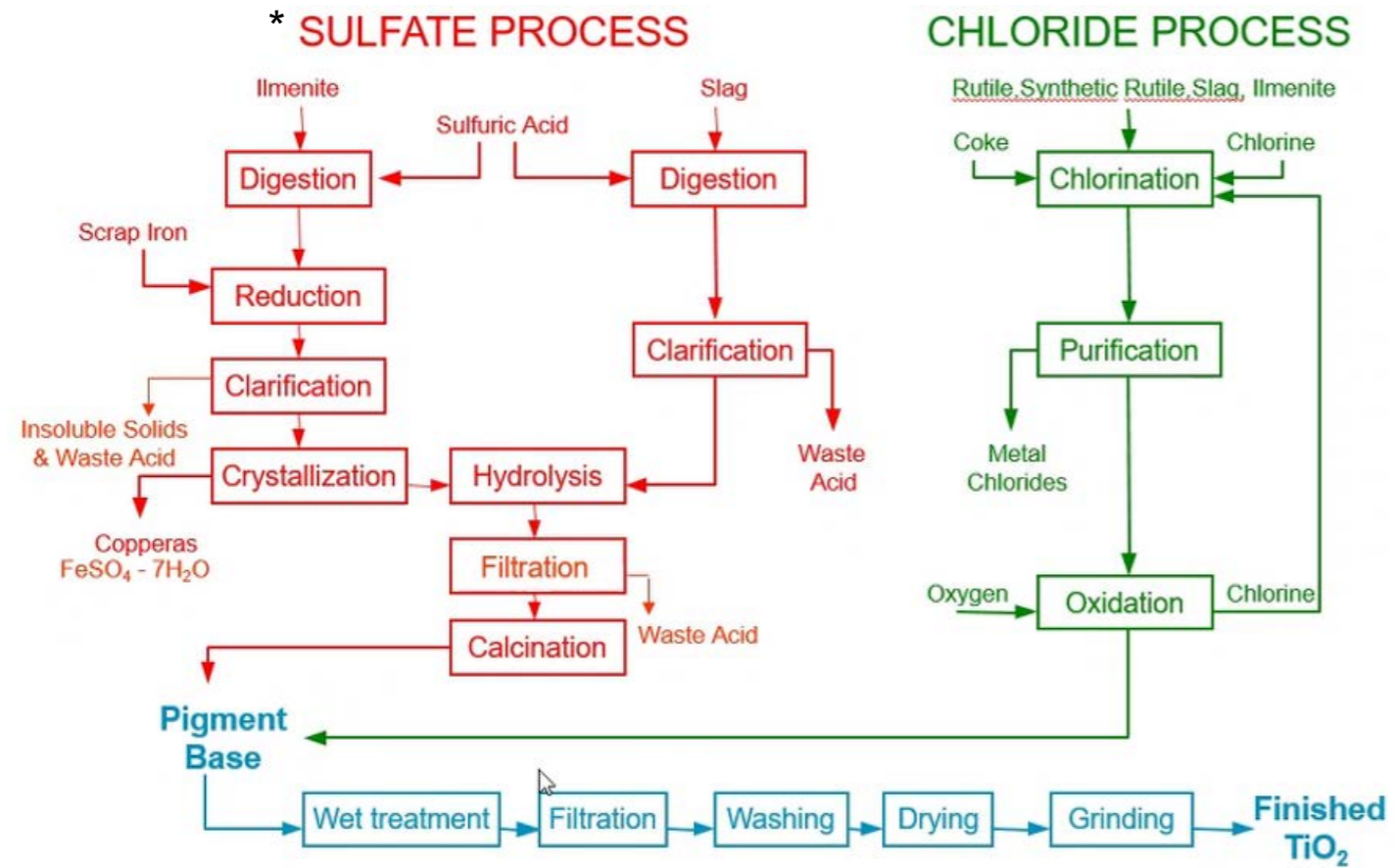
Pigments  
(86%)



Titanium Dioxide  
 $TiO_2$   
(59%)



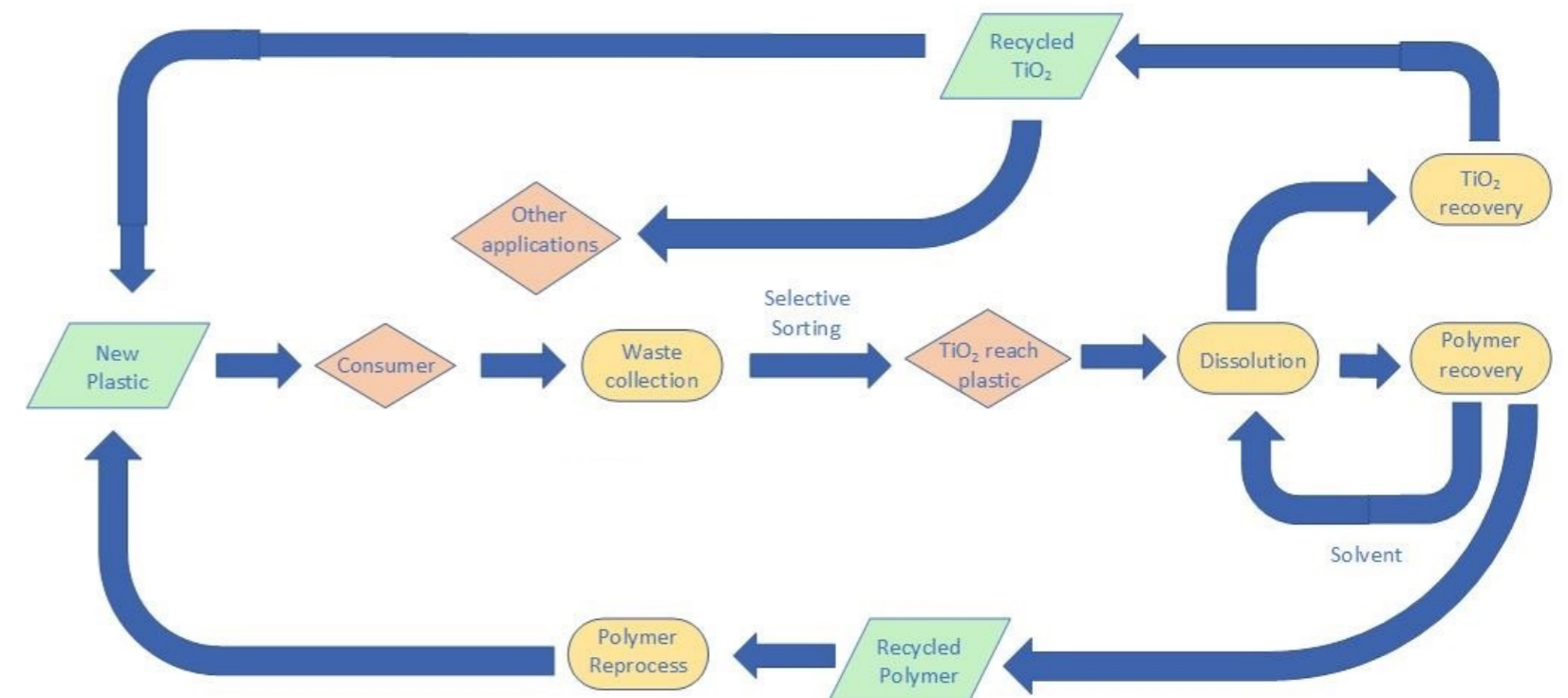
# CIRCULARITY TiO<sub>2</sub> PLASTICS



**SULFATE PROCESS**  
 Concentrated sulfuric acid  
 Solid and acid waste  
 117 MJ/kgTiO<sub>2</sub> \*\*

**CHLORIDE PROCESS**  
 Petroleum coke chlorine gas  
 Direct CO<sub>2</sub> emissions  
 106 MJ/kgTiO<sub>2</sub> \*\*

Compound	Carbon footprint	Price
	kgCO <sub>2</sub> e/kgcompound	€/kg (2021)
TiO <sub>2</sub>	4.90	2.6-3.2
Polymer	(LD)PE	1.2-1.3
	PVC	~1.2
	PS	~1.6



# PROCESS

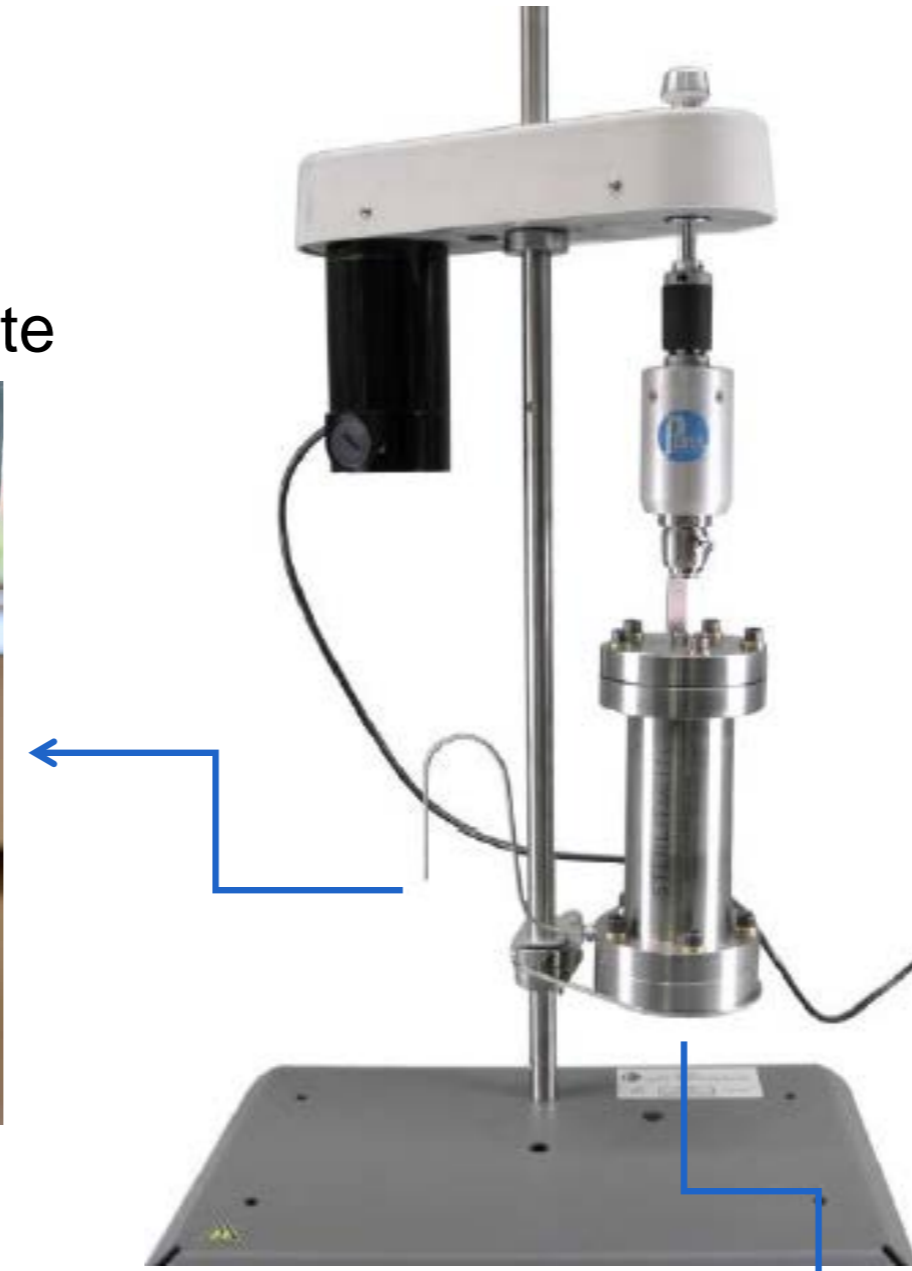
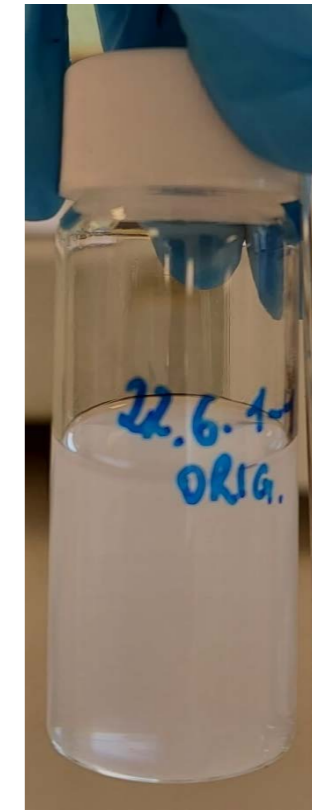
Solvent

Polymer containing  $\text{TiO}_2$



+

Dissolution



Set-up

(dead-end filtration)

Retentate



Permeate



Antisolvent



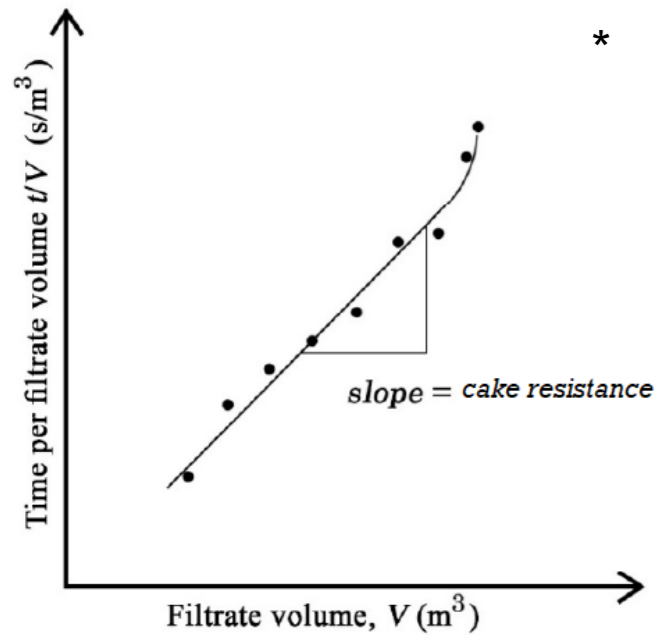
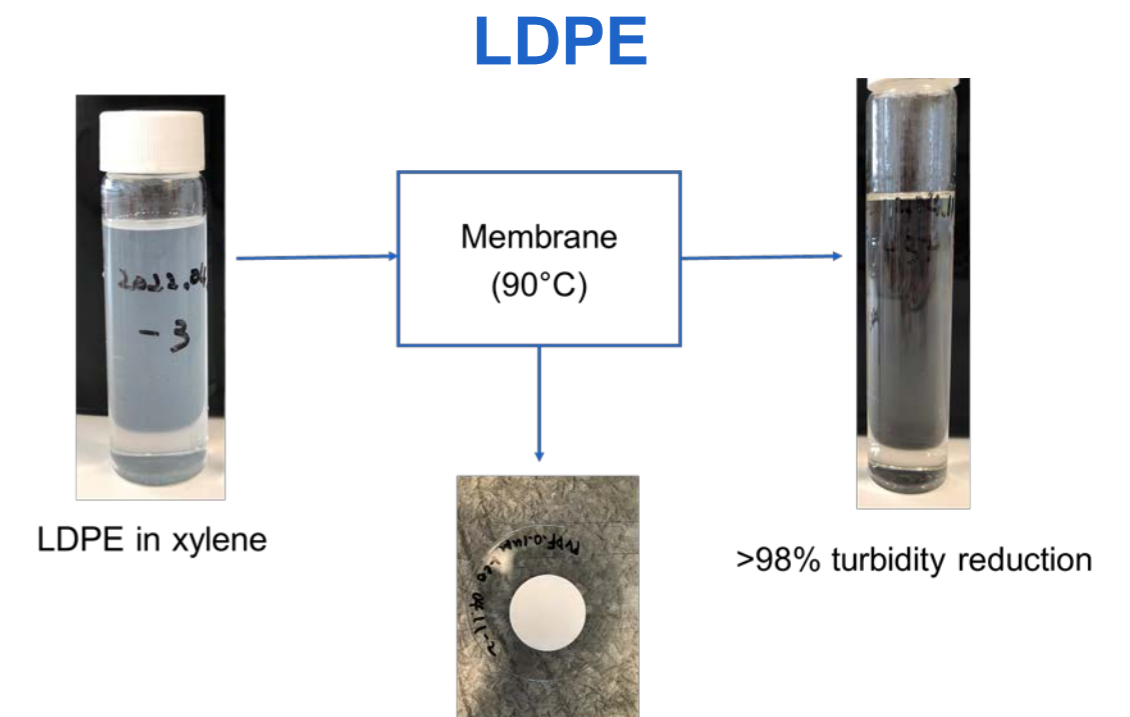
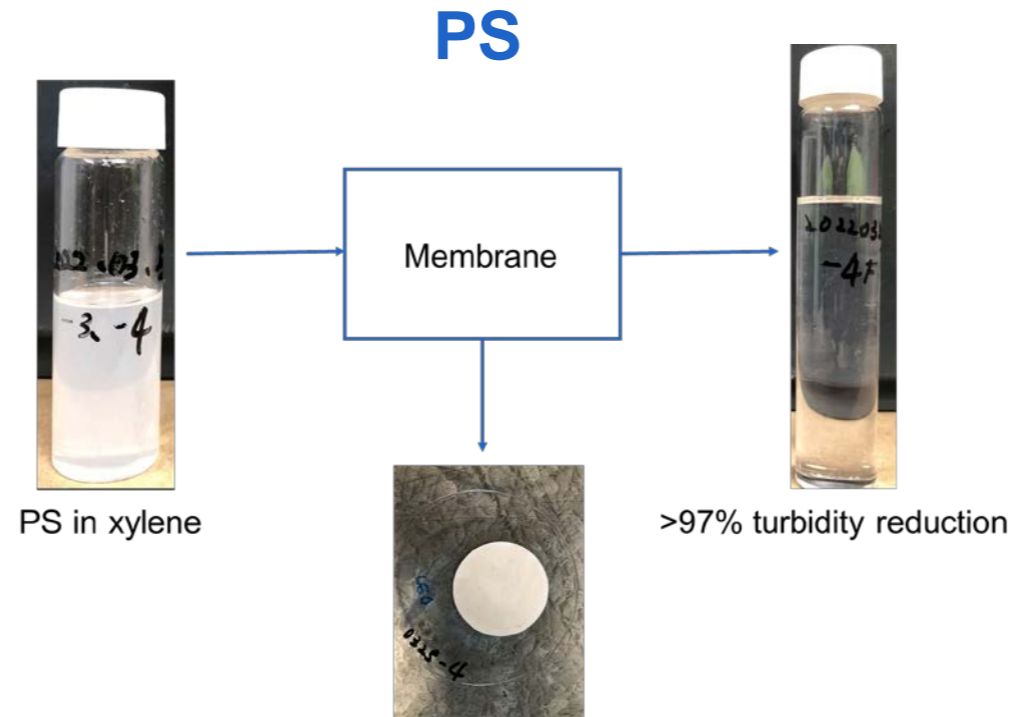
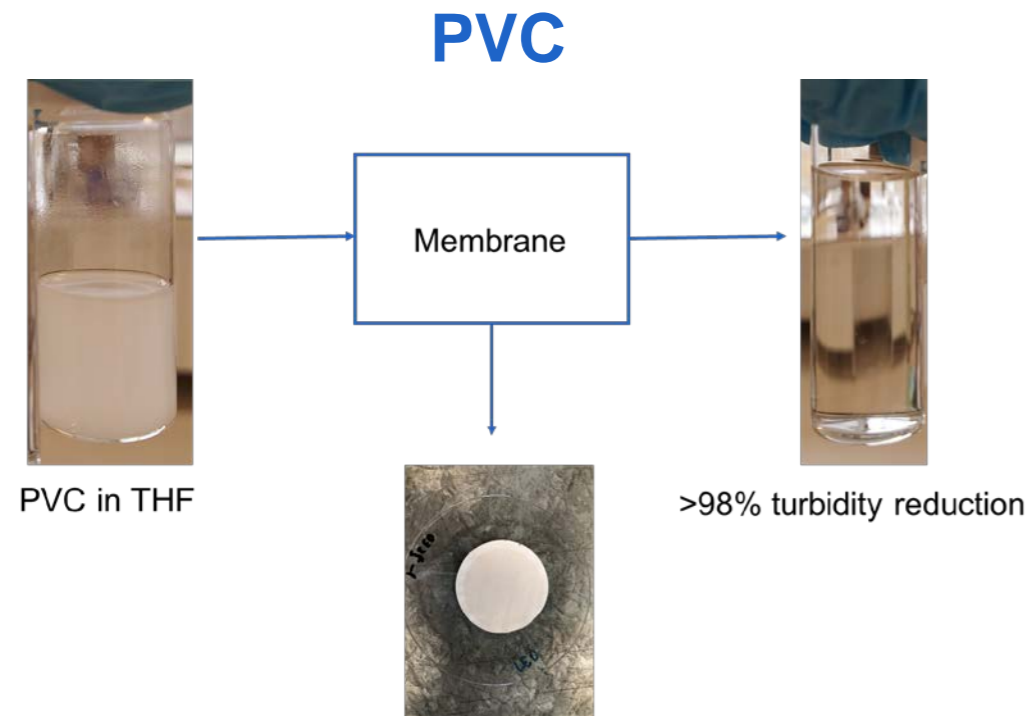
Precipitation



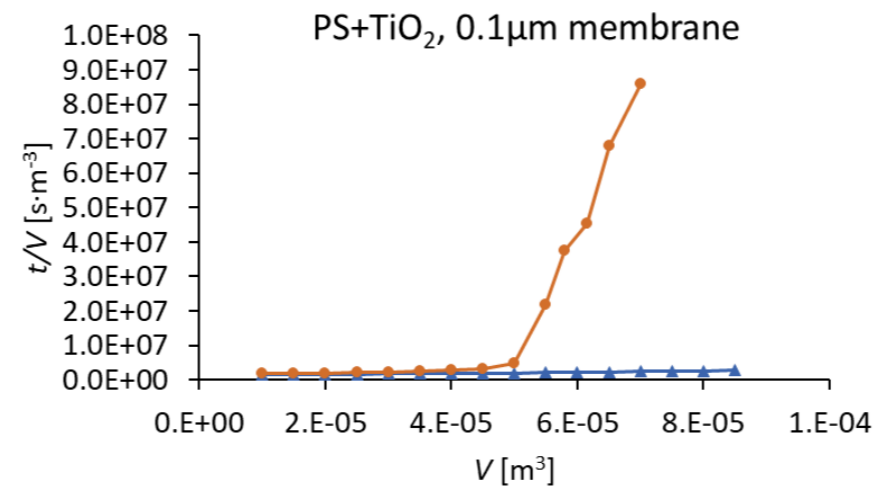
Recovered polymer

# TiO<sub>2</sub> SEPARATION

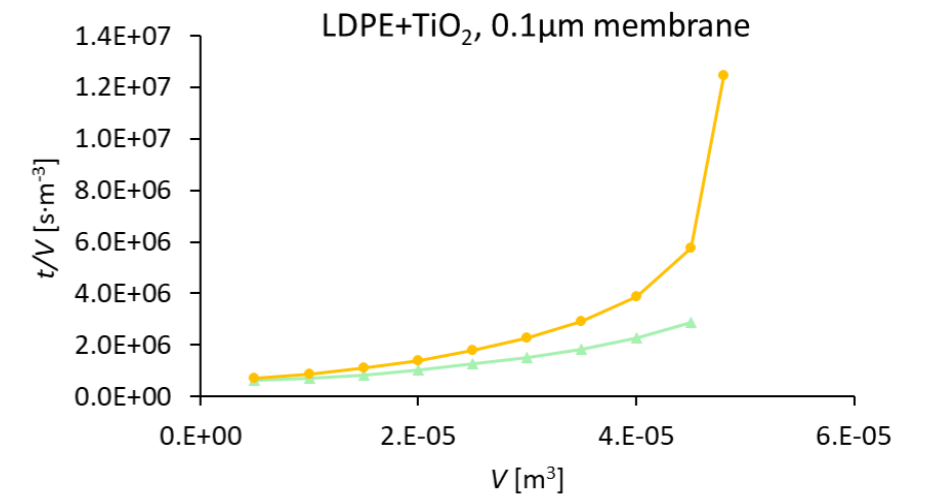
# MODEL SAMPLES



$$\frac{t}{V} = \frac{\mu_0 \alpha C_m}{2A^2 \Delta P} \cdot V + \frac{\mu_0 R_M}{A \Delta P}$$



- PS+TiO<sub>2</sub>, 5w% in xylene, 0.28w% TiO<sub>2</sub> in PS, 3 bar
- PS+TiO<sub>2</sub>, 5w% in limonene, 0.28w% TiO<sub>2</sub> in PS, 3 bar

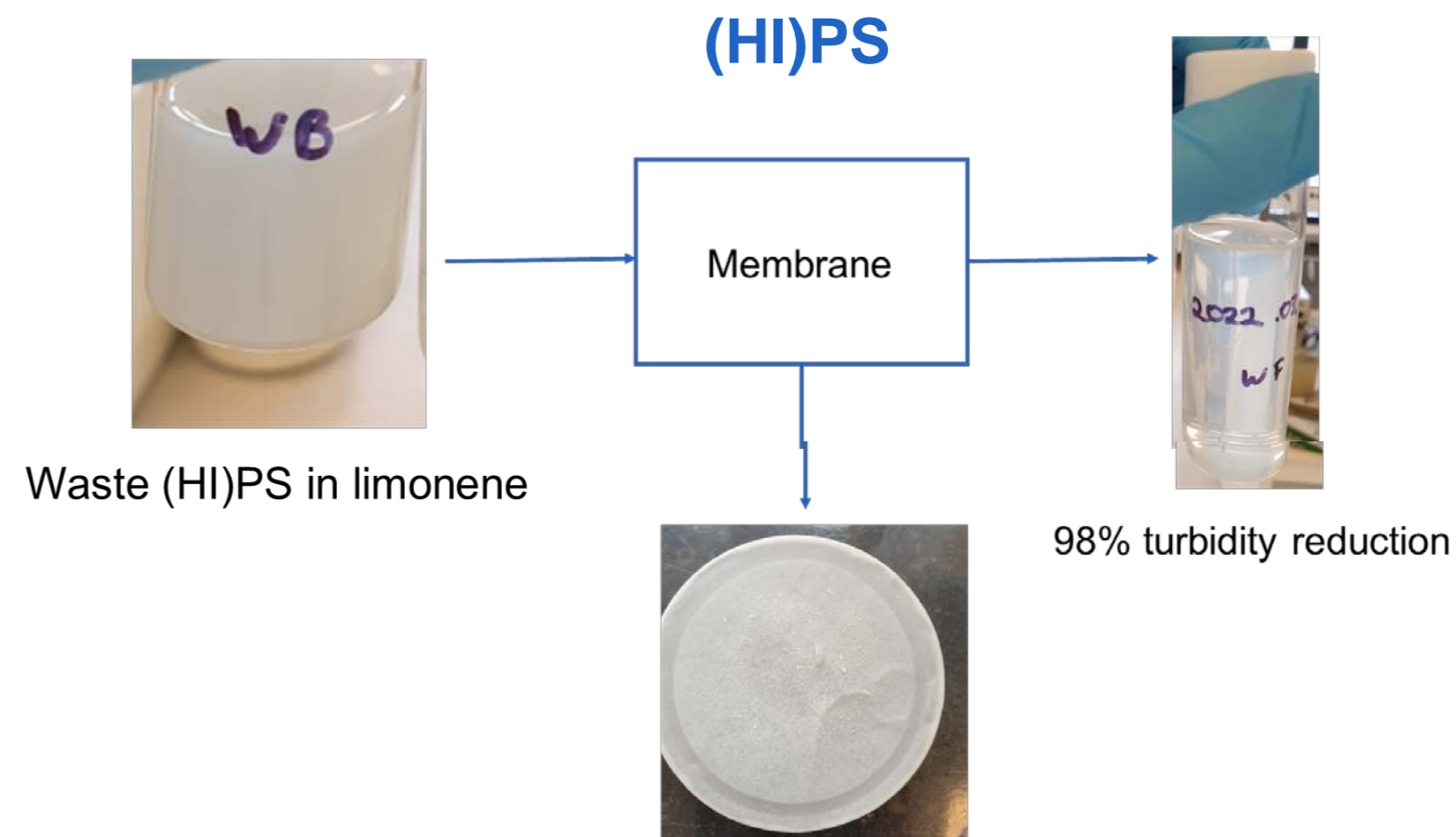
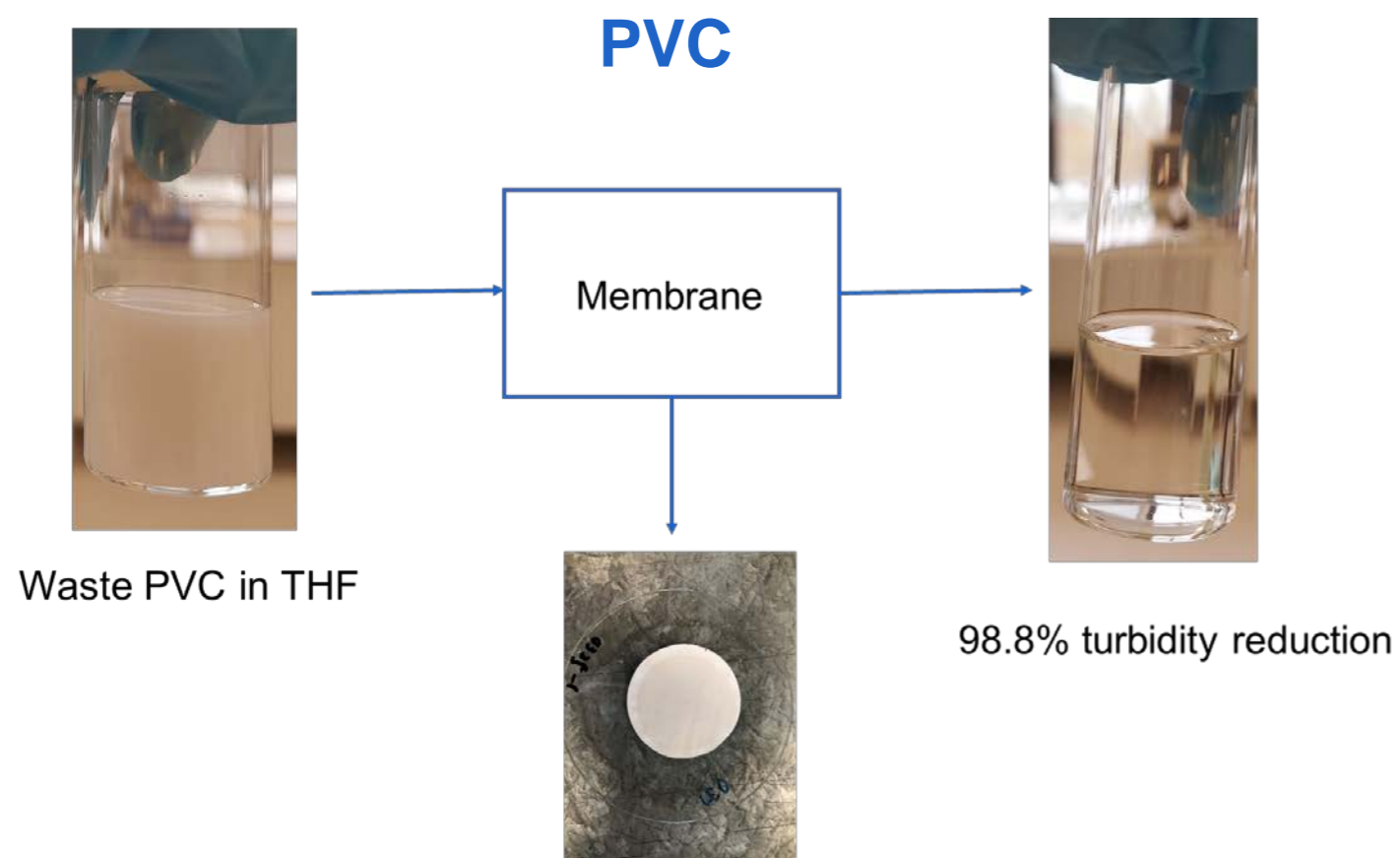


- LDPE+TiO<sub>2</sub>, 1w% in xylene, 0.24w% TiO<sub>2</sub> in LDPE, 3 bar, 90°C
- LDPE+TiO<sub>2</sub>, 1w% in limonene, 0.24w% of TiO<sub>2</sub> in LDPE, 3 bar, 90°C

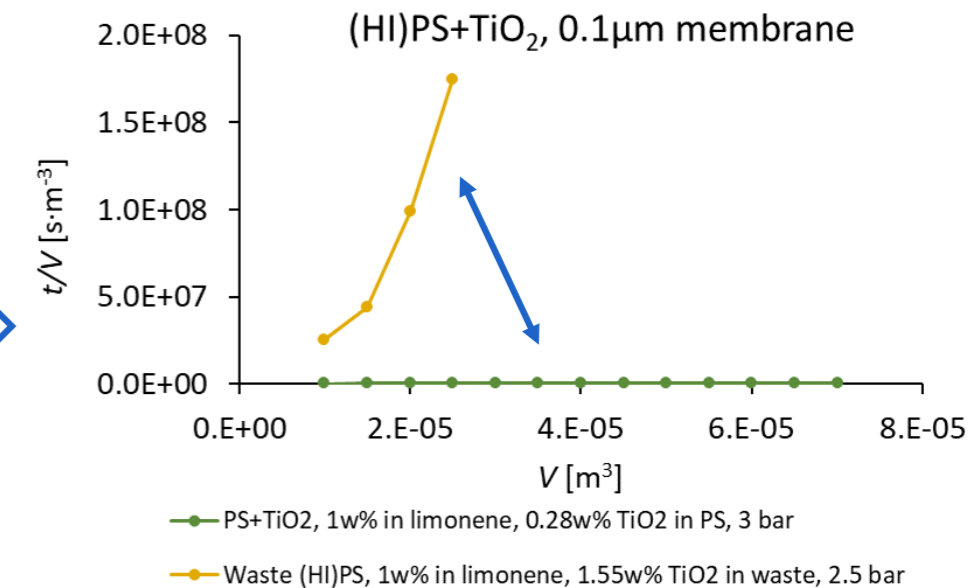
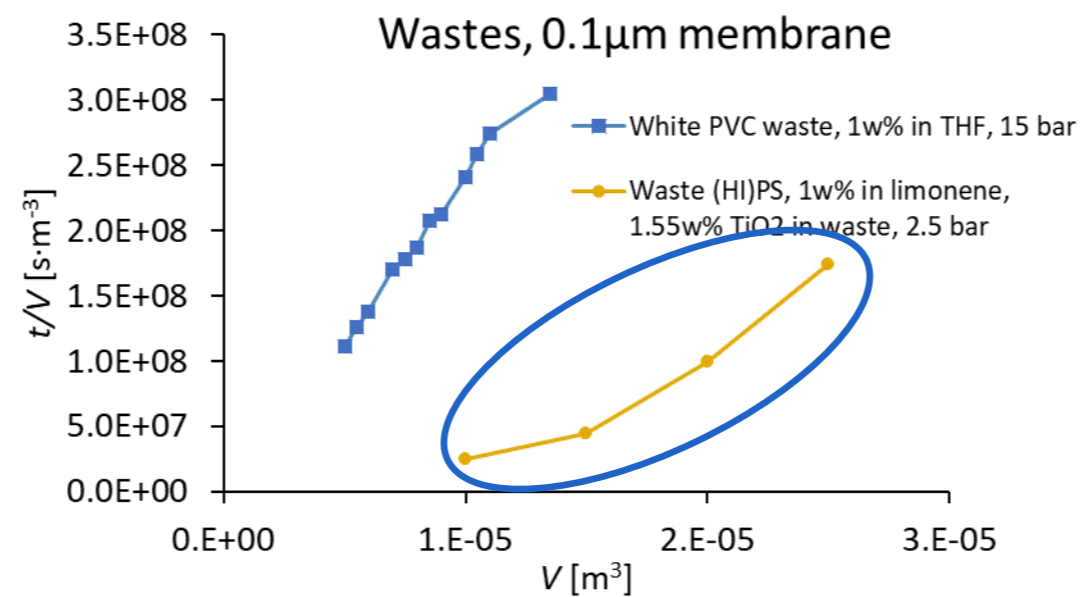
Solvent influences the flow rate and cake resistance



# WASTE SAMPLES



Waste samples significantly higher cake resistance



# RECOVERY

# COMPOUNDS RECOVERY

## Polymer



Permeate

Precipitation bath  
(antisolvent)



Recovered  
polymer

Almost pure polymer sheet  
is obtained  
(4w% (anti)solvent trace)

## TiO<sub>2</sub>



Carbon black+TiO<sub>2</sub>

Oven



TiO<sub>2</sub>

TiO<sub>2</sub> can be separated from  
organic compounds

# CONCLUSIONS & FUTURE PROSPECTIVE

- **Solvent-based** recycling **promising** route for plastic recycling
- **Concurrent recovery** of polymer and  $\text{TiO}_2$  is possible
- Filtration efficiency depends on: solvent, concentration, polymer, additives
  
- Optimization of the process required to treat real waste streams
- Assess recovered polymer properties
- Explore alternative routes for  $\text{TiO}_2$  purification

# Thank you for your attention!

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Ghent University



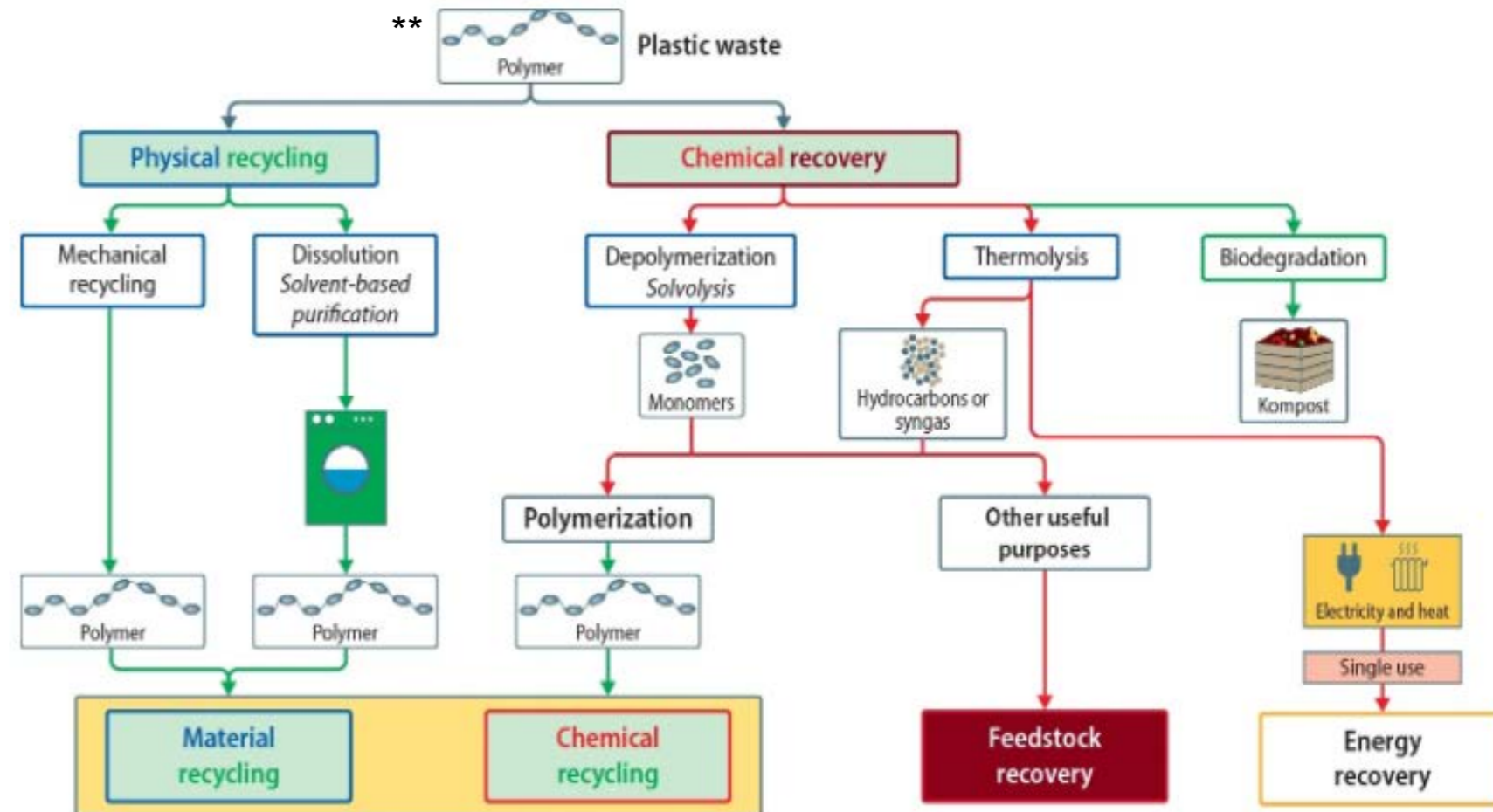
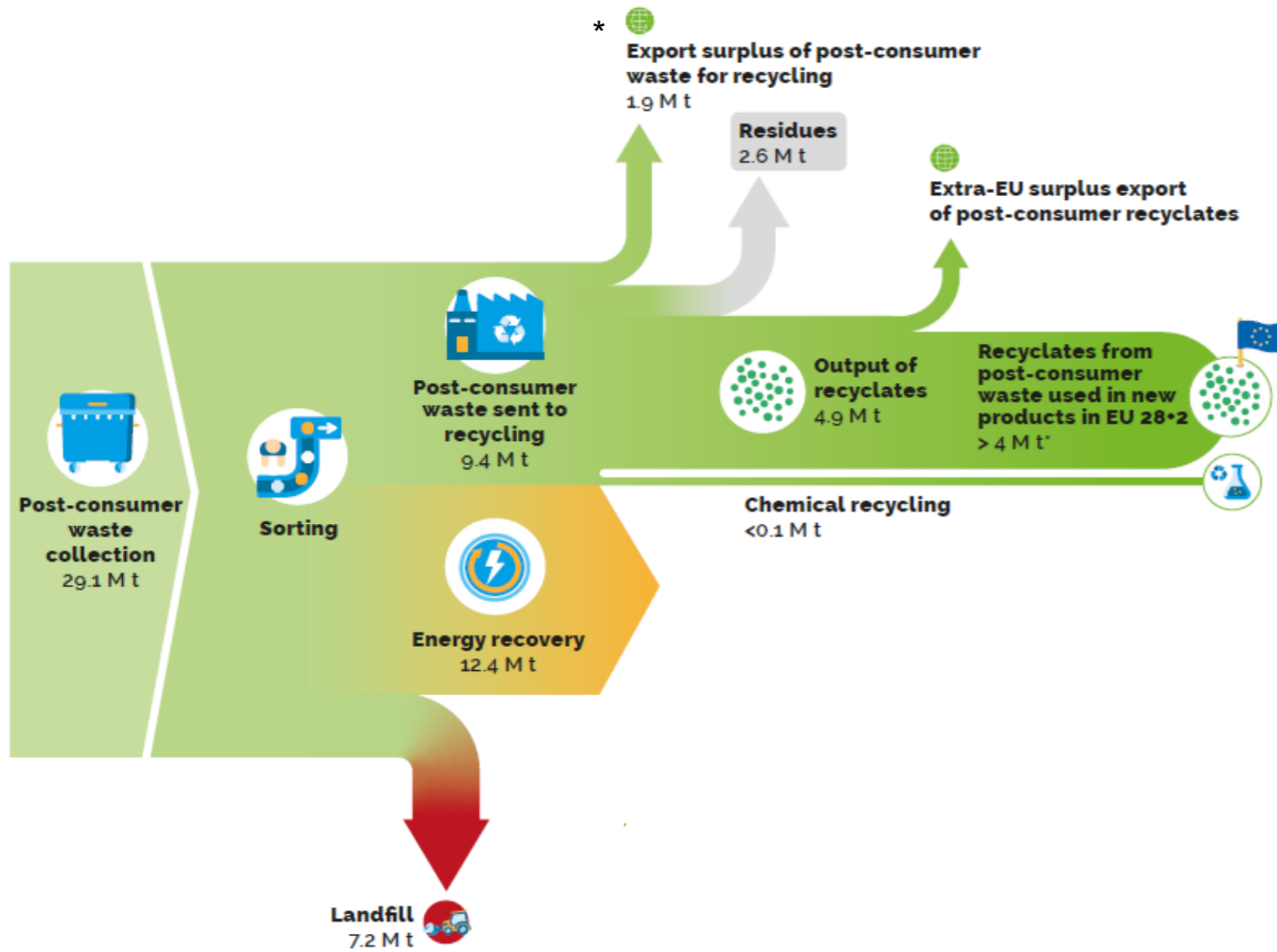
@ugent



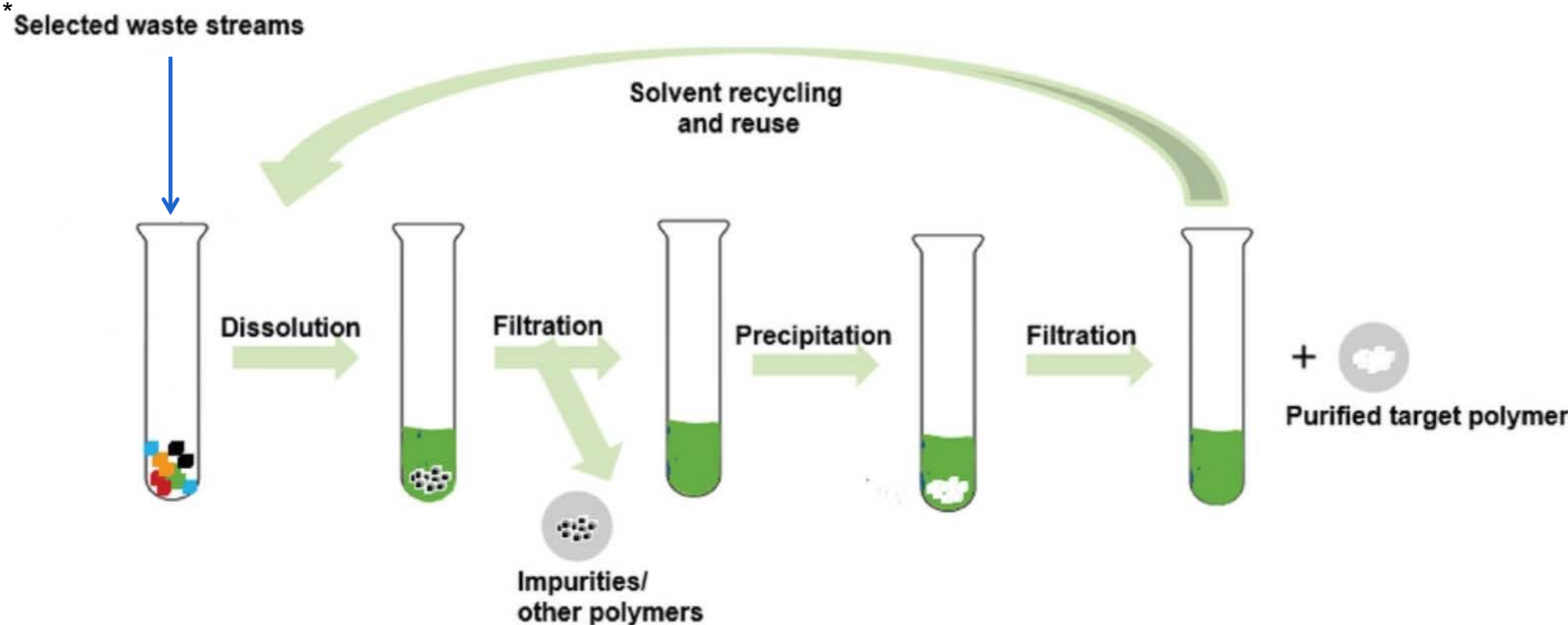
Ghent University

[www.lcpe.ugent.be](http://www.lcpe.ugent.be)

# PLASTICS RECYCLING RATES



# DISSOLUTION-PRECIPITATION PROCESS

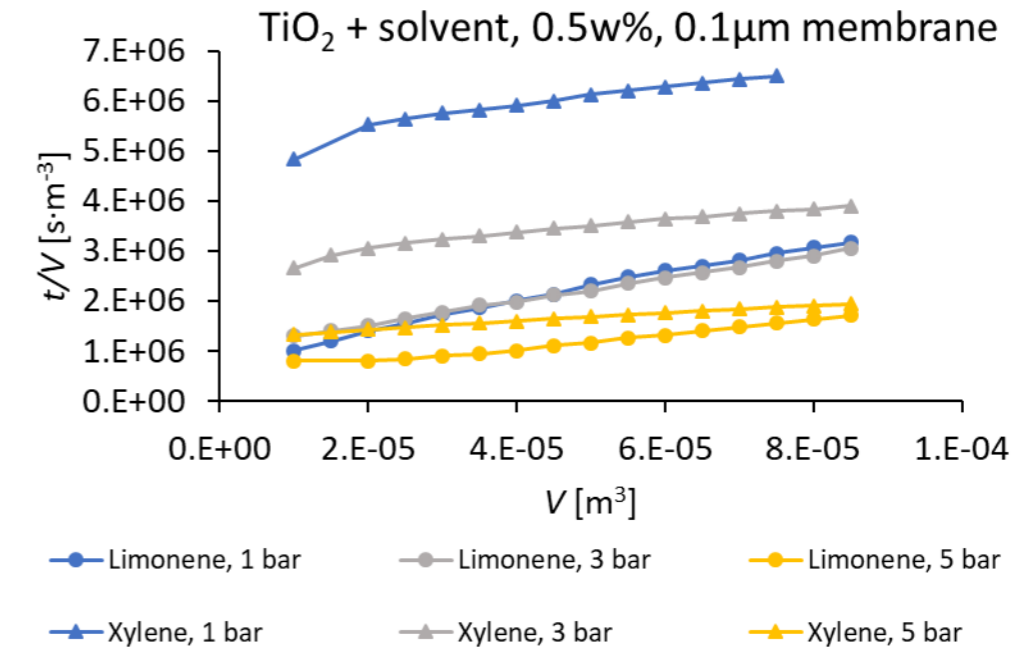


\*Figure adapted from Mangold (2022), The Frontier of Plastics Recycling: Rethinking Waste as a Resource for High-Value Applications.

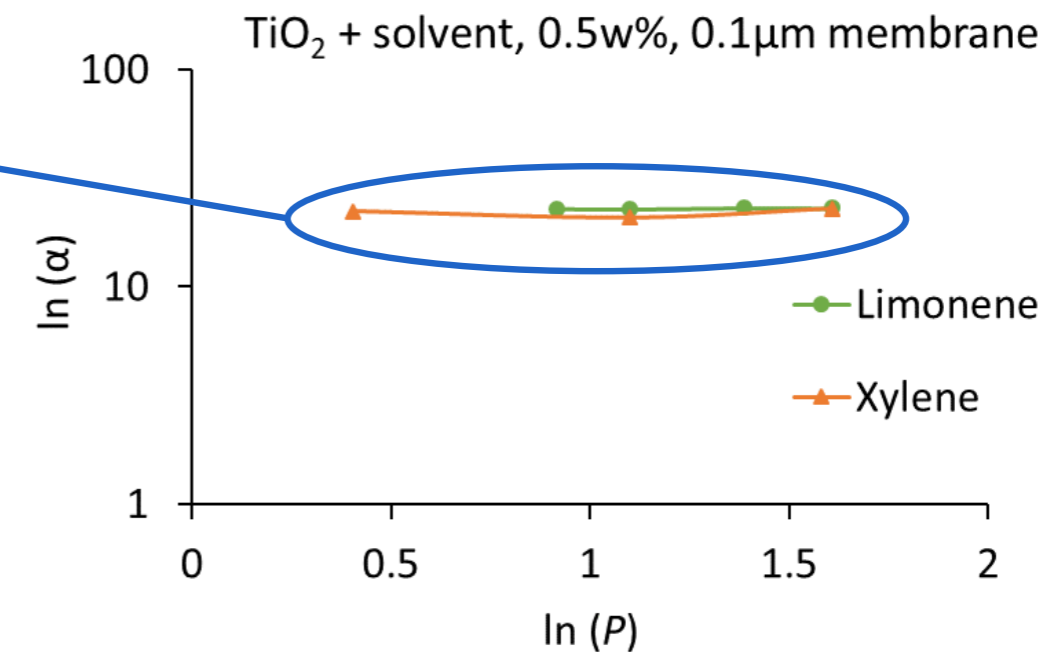
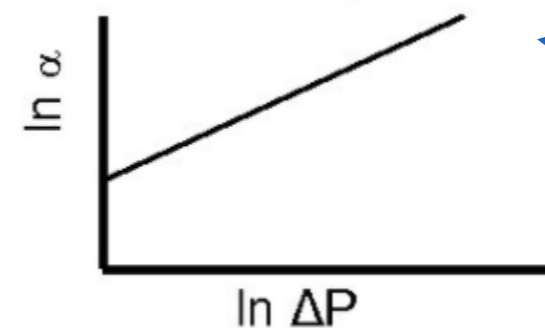
# FILTRATION CURVES

## Pure TiO<sub>2</sub> filtration (R-350)

Sample	Solvent	Pressure (bar)	Turbidity reduction (%)
TiO <sub>2</sub> 0.5wt%	Xylene	1	100
		3	100
		5	100
	Limonene	1	100
		3	100
		5	100

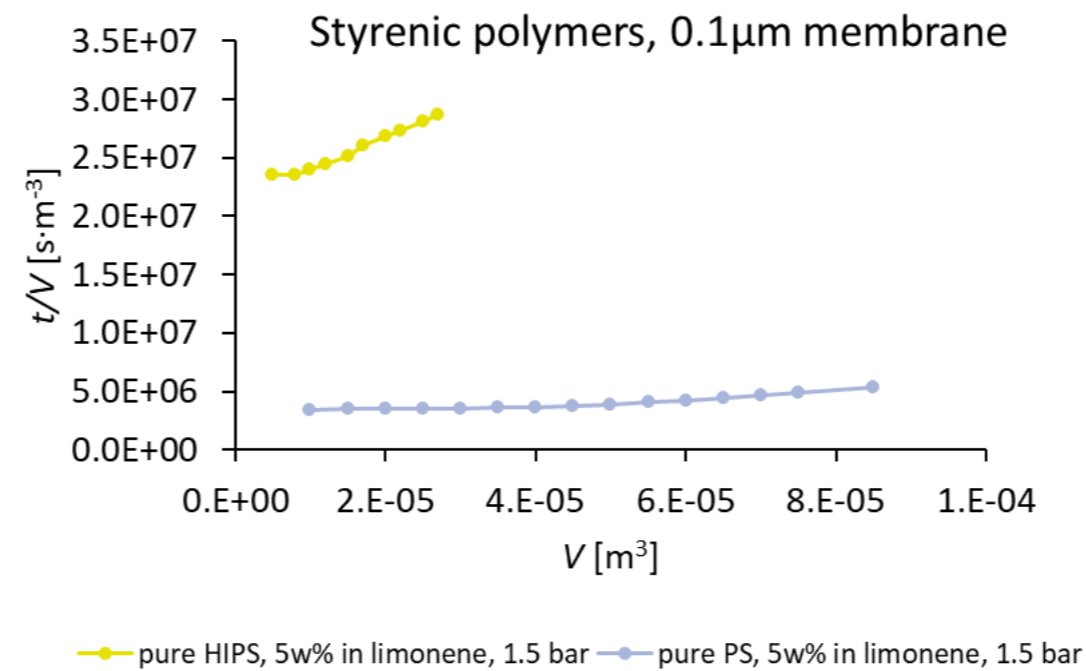
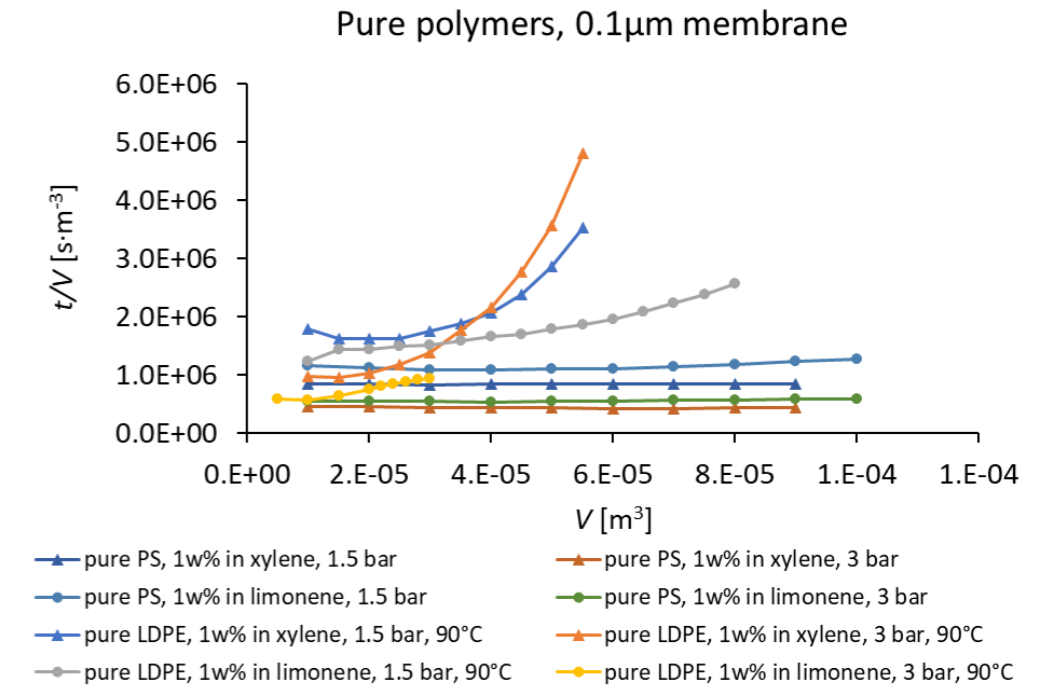
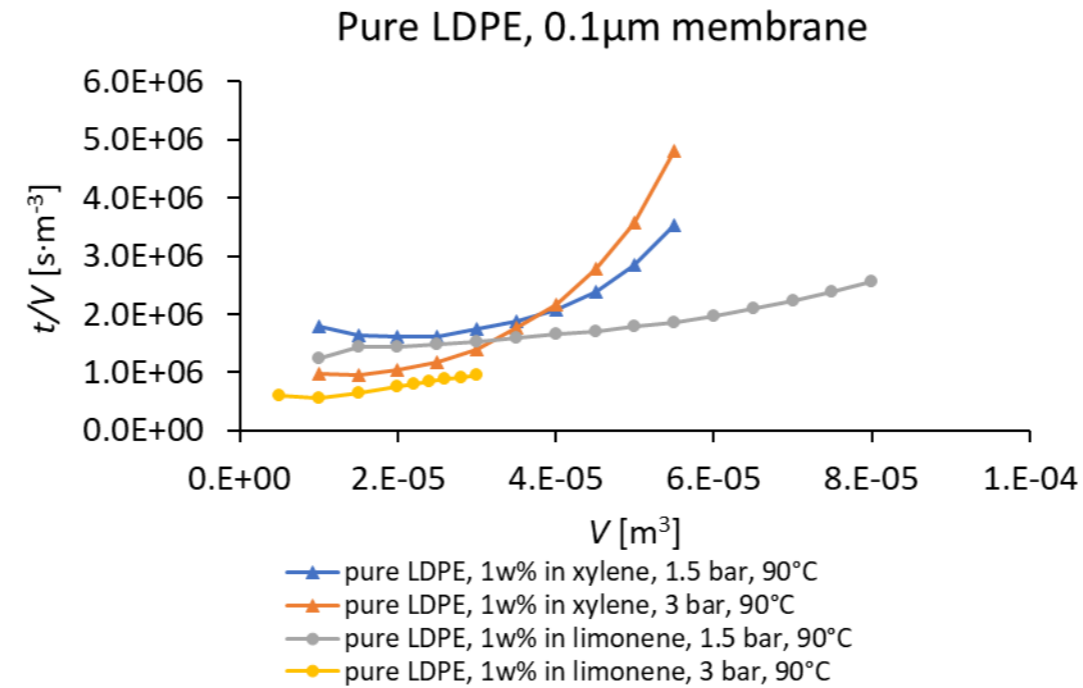
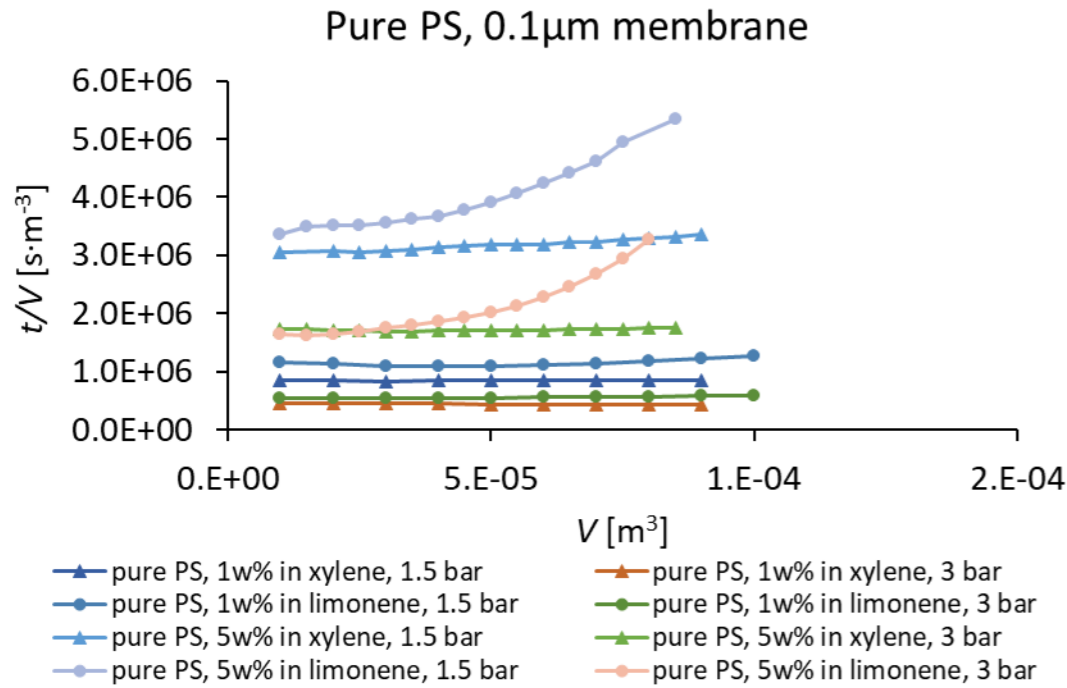


No significant compressibility





# PURE POLYMER FILTRATIONS



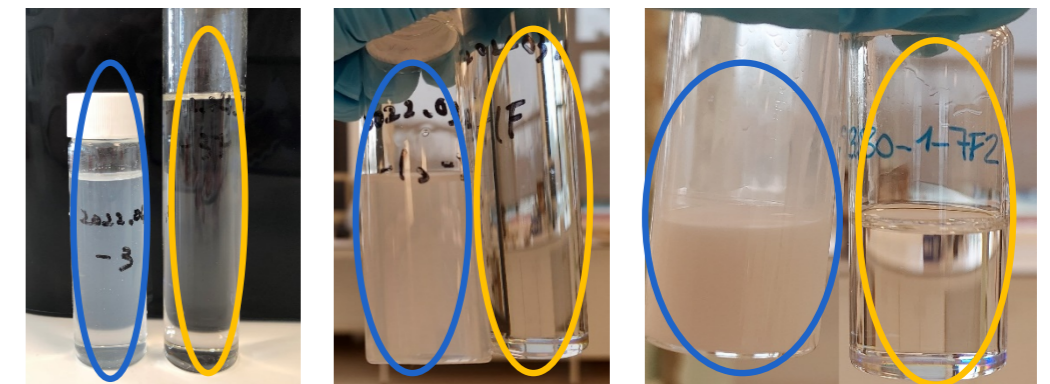
# FILTRATION EXPERIMENTS

## PS+TiO<sub>2</sub> (R-350)



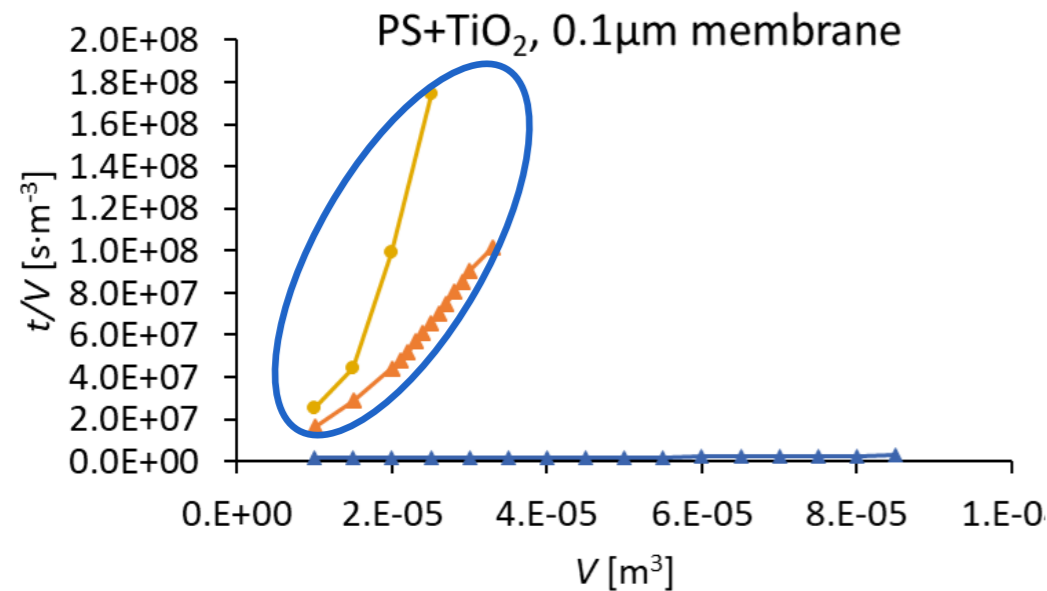
Sample	Solvent	Pressure (bar)	Turbidity reduction (%)	Polymer retention (%)
PS+TiO <sub>2</sub> , 5w% solution (0.28w% TiO <sub>2</sub> in PS)	Xylene	1.5	98.6	5.2
		3	99.5	2.6
	Limonene	1.5	97.5	1.2
		3	98.7	3.4
PS+TiO <sub>2</sub> , 1w% solution (0.28w% TiO <sub>2</sub> in PS)	Xylene	1.5	96.7	6.2
		3	96.8	7.7
	Limonene	1.5	96.7	1.1
		3	96.6	0.0
PS+TiO <sub>2</sub> , 7w% solution (10w% TiO <sub>2</sub> in PS)	Xylene	9	98.9	5.4

TiO<sub>2</sub> can be easily separated from pure PS by filtration

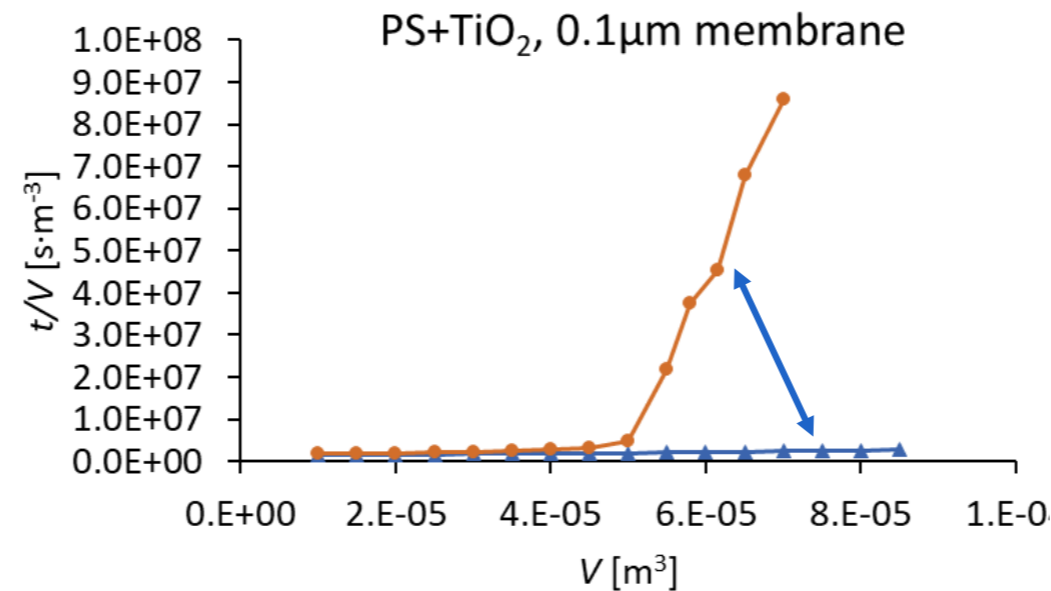


# FILTRATION CURVES

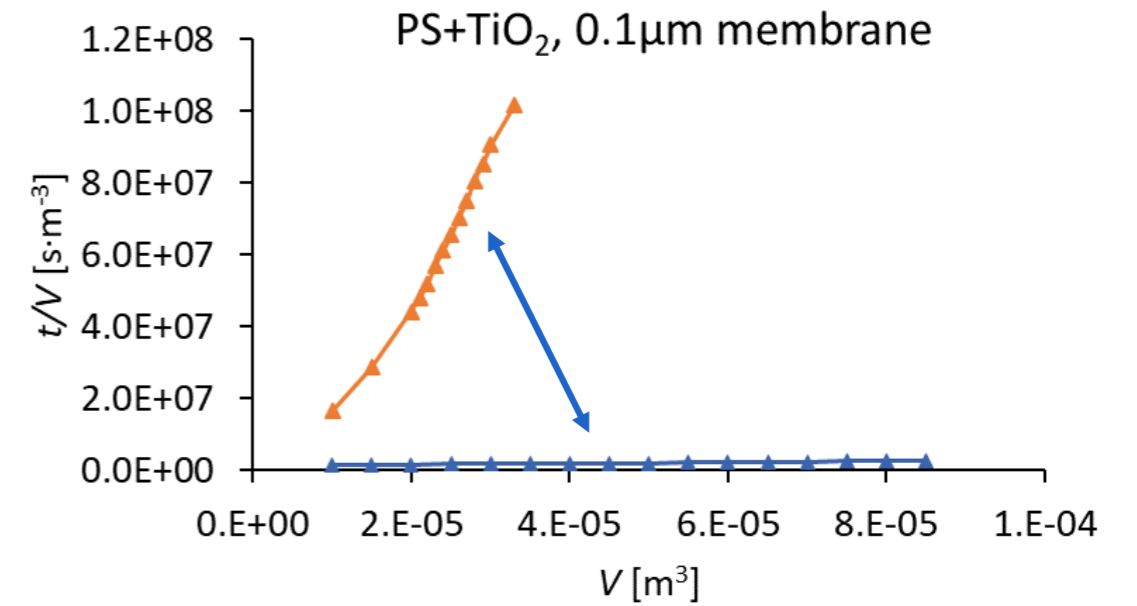
## Styrenic polymers



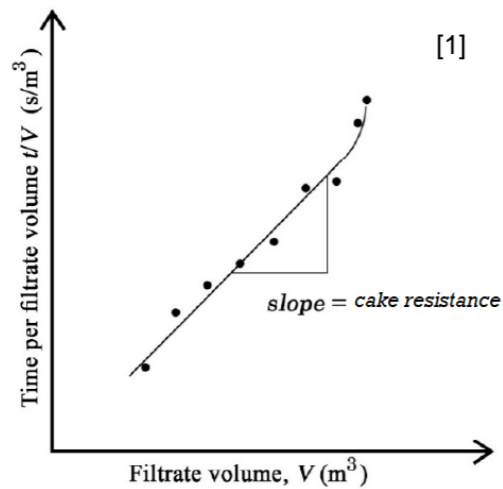
- ▲ PS+TiO<sub>2</sub>, 7w% in xylene, 10w% TiO<sub>2</sub> in PS, 9 bar
- ▲ PS+TiO<sub>2</sub>, 5w% in xylene, 0.28w% TiO<sub>2</sub> in PS, 3 bar
- Waste (HI)PS, 1w% in limonene, 1.55w% TiO<sub>2</sub> in waste, 2.5 bar



- ▲ PS+TiO<sub>2</sub>, 5w% in xylene, 0.28w% TiO<sub>2</sub> in PS, 3 bar
- PS+TiO<sub>2</sub>, 5w% in limonene, 0.28w% TiO<sub>2</sub> in PS, 3 bar



- ▲ PS+TiO<sub>2</sub>, 7w% in xylene, 10w% TiO<sub>2</sub> in PS, 9 bar
- ▲ PS+TiO<sub>2</sub>, 5w% in xylene, 0.28w% TiO<sub>2</sub> in PS, 3 bar



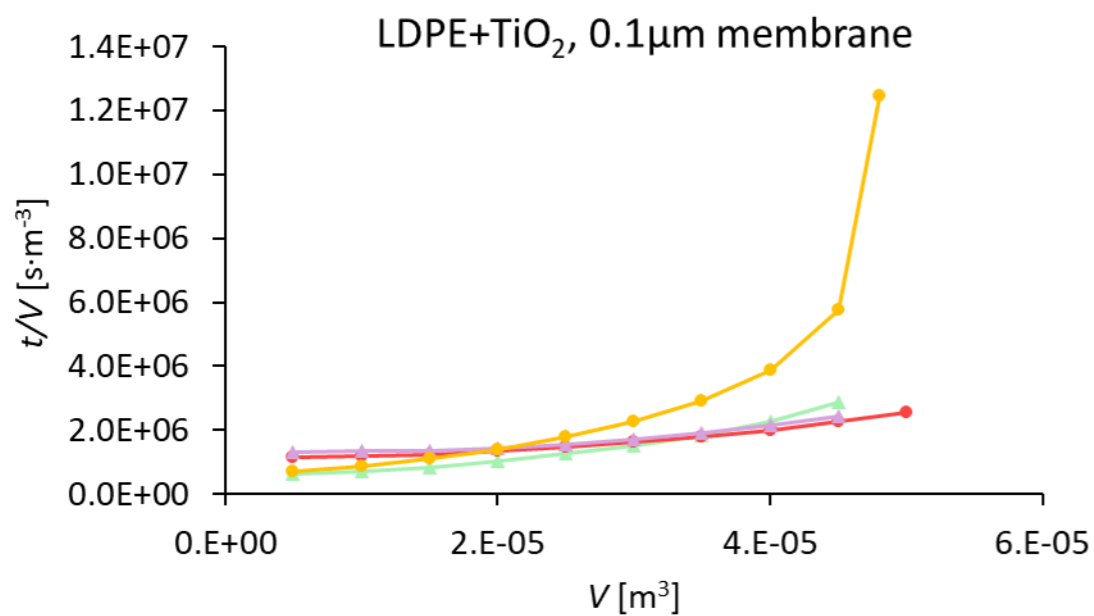
- Waste sample behaves similar to high concentration of pure PS+TiO<sub>2</sub> solution
- Solvent influences cake resistance
- Increasing concentration, cake resistance increases significantly

# FILTRATION EXPERIMENTS

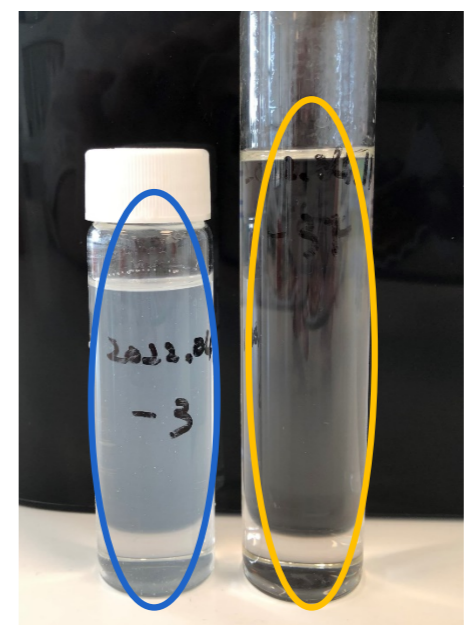
## LDPE+TiO<sub>2</sub> (R-350)



Sample	Solvent	Pressure (bar)	Turbidity reduction (%)	Polymer retention (%)
LDPE+TiO <sub>2</sub> , 1w% solution (0.24w% TiO <sub>2</sub> in LDPE)	Xylene	1.5	98.6	1.3
		3	99.5	0.9
	Limonene	1.5	97.5	0.8
		3	98.7	0.3



TiO<sub>2</sub> can be easily separated from pure LDPE by filtration

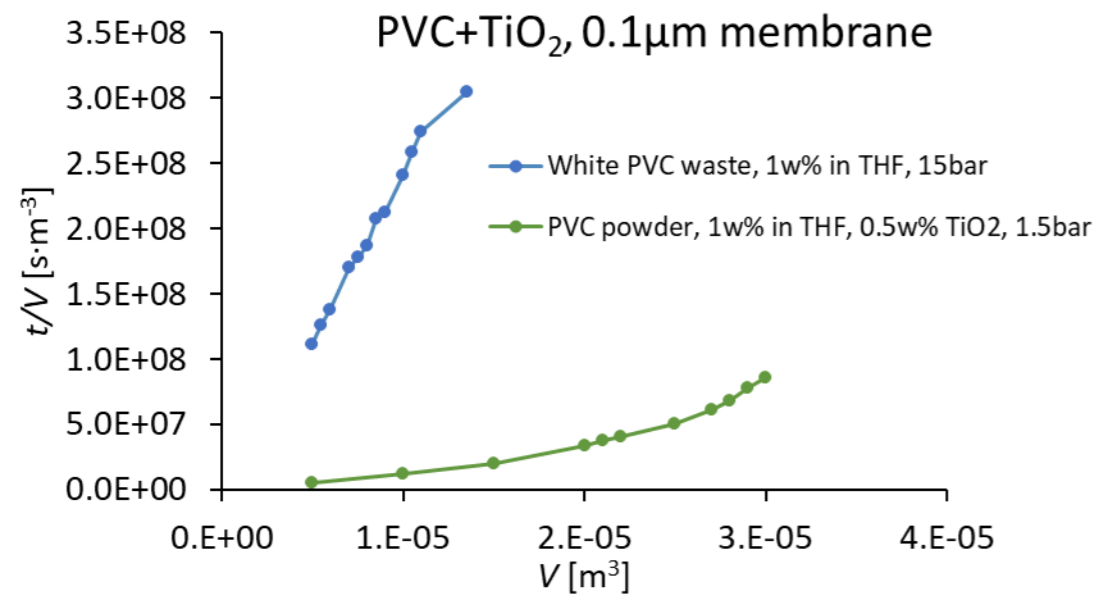


# FILTRATION EXPERIMENTS

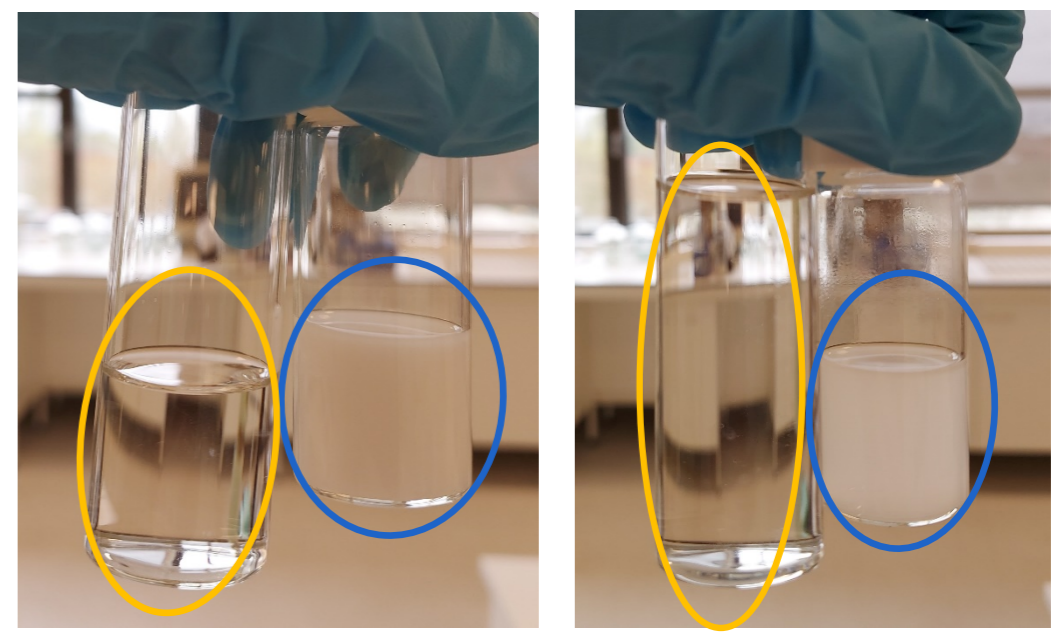
## PVC (Deceuninck)



Sample	Solvent	Pressure (bar)	Turbidity reduction (%)	Polymer retention (%)
PVC, 1w% (0.5w% TiO <sub>2</sub> in PVC+additives)	THF	1.5	98.8	7.9
Waste PVC, 1w%		15	99.9	76.3



TiO<sub>2</sub> can be separated from PVC by filtration  
(also for waste sample\*)



Waste PVC

PVC+additives

\* = optimization needed