

# PL<sup>♻️</sup>ST2bCLE<sup>♻️</sup>NED

## PART I

**KER I:** Novel polymer recycling technology based on dissolution

**KER II:** Novel antimony oxide (ATO) and bromine (BR) recycling recovery technology



This Project has received funding from the European Union's Horizon 2020 Research and Innovation Programme under Grant Agreement N. 821087

**Annemieke van de Runstraat**

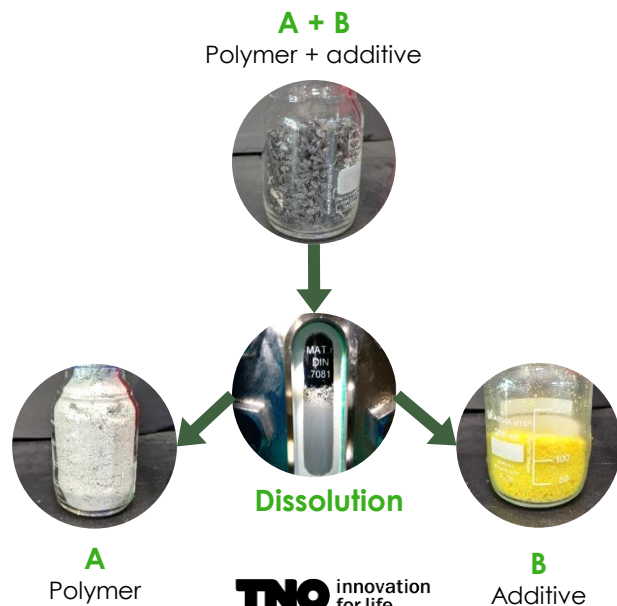
Project Manager & Team leader – TNO

**Sebastian Reinhardt**

Scientist - Fraunhofer ICT



# KER I: DISSOLUTION



## Main characteristics

- Use a single low-boiling solvent above the boiling point
- Pressurised system
- Dissolve the polymer in the plastic
- Thereby release additives
- Remove additives from the polymer
- Recover polymer, additives and recycle solvent

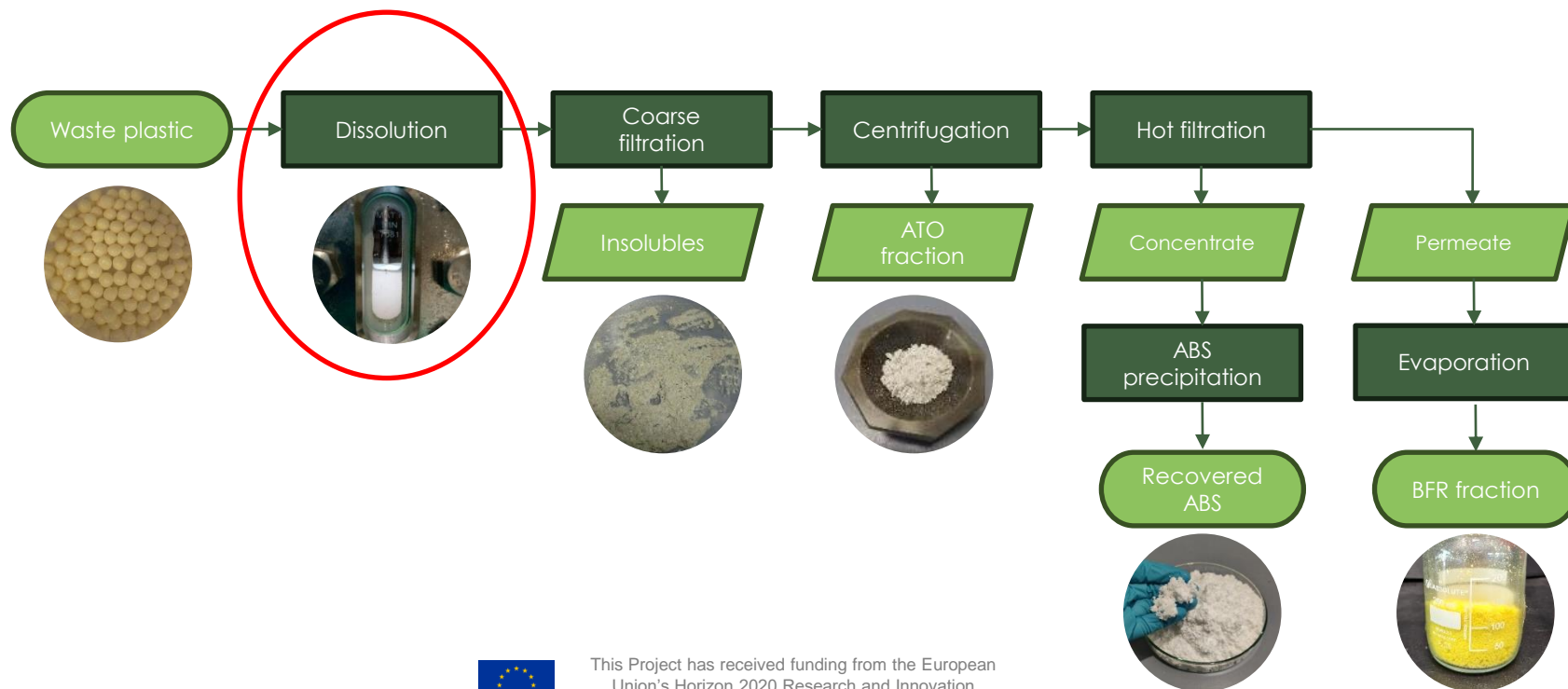
## Main benefits

- Energy efficient process
- Potential for high-quality polymers: value retention
- Potential to also recover additives for recycling



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## PLAST2bCLEANED DISSOLUTION BASED PROCESS SUMMARY



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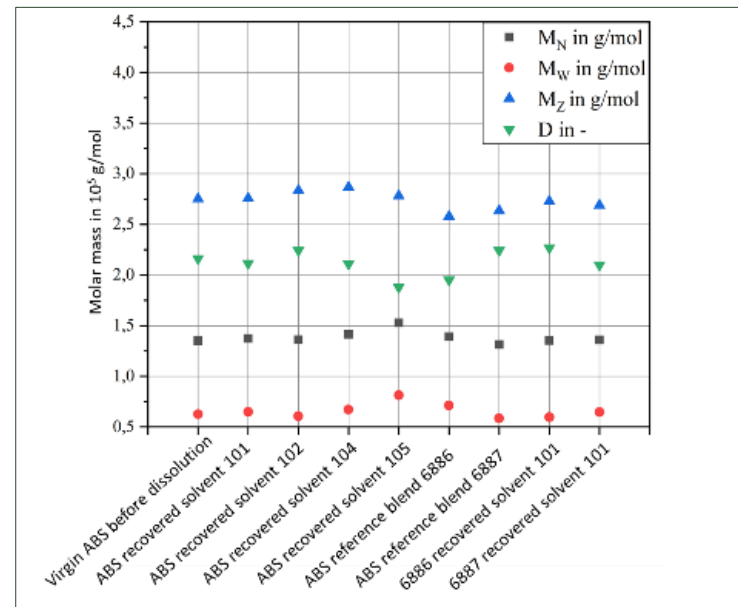
# KER I: DISSOLUTION OF ABS

## Dissolution tests

- The polymer is not changed during dissolution, chain length is not affected by the process
- Additives such as Bromine flame retardants dissolve and antimony trioxide is suspended in solution
- Dissolution trials on real waste plastics in kg scale showed an insoluble fraction of ~2%.



Aluminium foil, paper, metals,  
insoluble plastics from coarse filtration



SEC of ABS samples before and after dissolution



## PLAST2bCLEANED DISSOLUTION BASED PROCESS SUMMARY

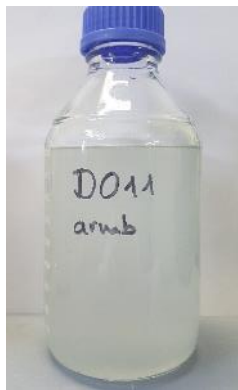


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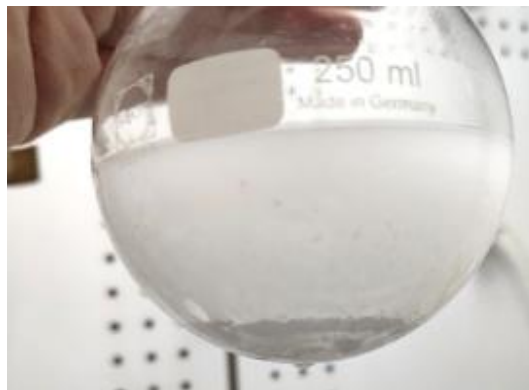


## KER II: REMOVAL OF ANTIMONY TRIOXIDE

- Antimony trioxide (ATO) will not dissolve → particles are suspended
- Due to high density ( $5.2 \text{ g/cm}^3$ ) ATO shows sedimentation behavior (but low particle size [ $0.9 - 1.2 \mu\text{m}$ ] makes for slow sedimentation)



Dissolved HiPS


Dissolved HiPS  
containing ATO


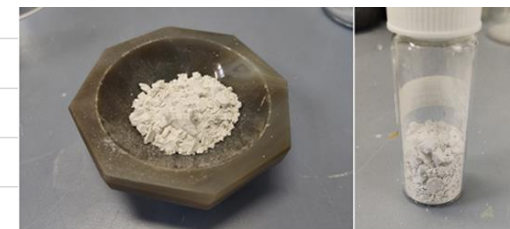
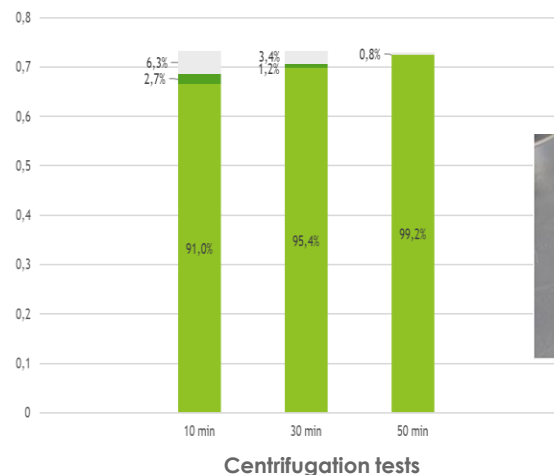
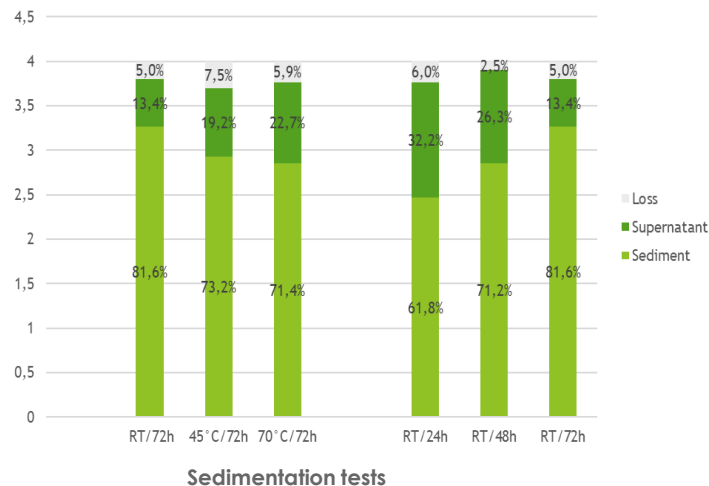
Sedimentation after 72 hours


Supernatant of centrifugation experiment  
after 10 min, 30min, 50min


## KER II: REMOVAL OF ANTIMONY TRIOXIDE

### Removal of antimony trioxide by density separation

- Density separation tests along multiple approaches (sedimentation, centrifugation, decanter centrifuge) showed good separation on reference substrate (>99% removal)



Recovered ATO fraction  
>90% ATO  
<0,1% Br



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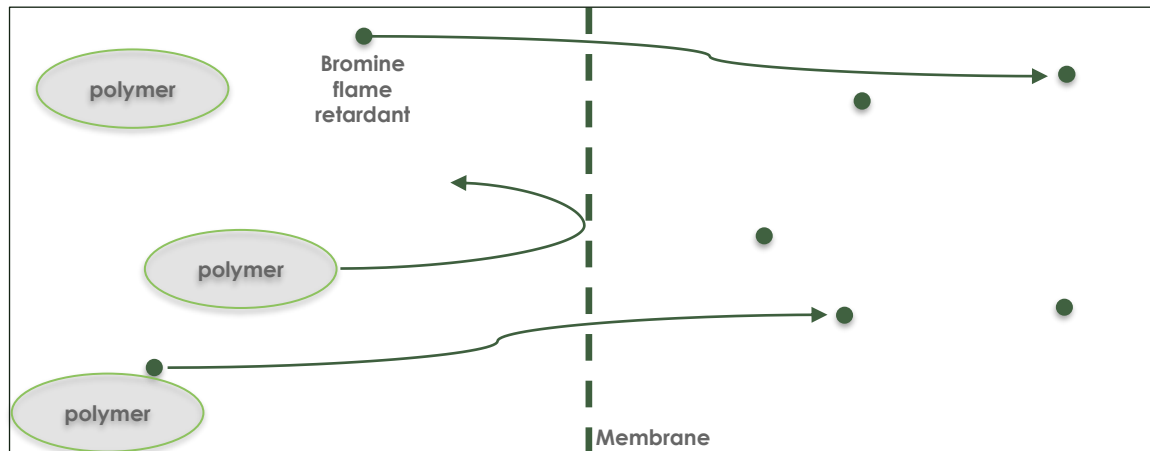


## PLAST2bCLEANED DISSOLUTION BASED PROCESS SUMMARY

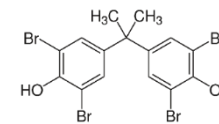


## KER II: REMOVAL OF BROMINE FLAME RETARDANT

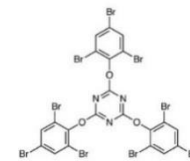
- Dissolved **bromine flame retardants (BFRs)** show molar masses in magnitude of **~1000 g/mol**.
- Dissolved **ABS** has average weighted mass Mw between **100.000 – 300.000 g/mol**



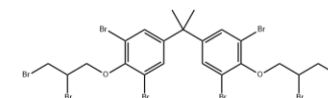
- BFR pass through membrane while larger polymer gets rejected



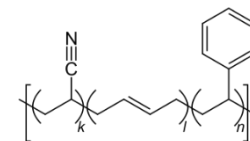
TBBPA Mw = 544g/mol



TTBP-TAZ Mw = 1067g/mol



TBBPA-DBPE Mw = 944g/mol



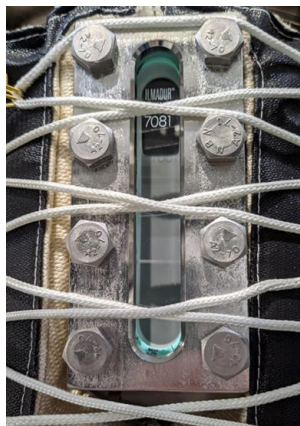
ABS = ~100.000 -300.000 g/mol



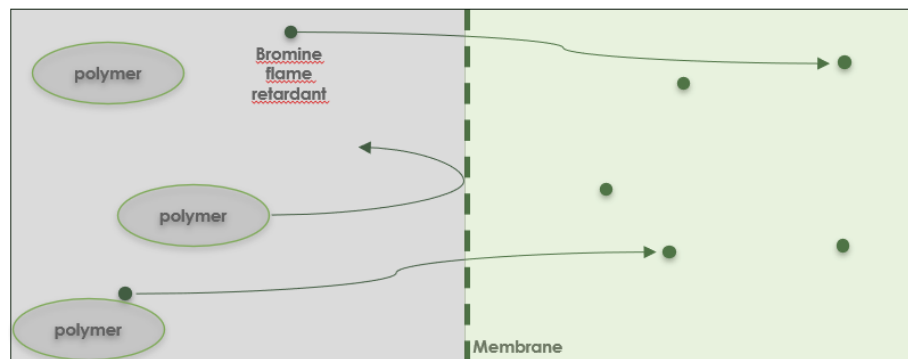
# KER II: REMOVAL OF BROMINE FLAME RETARDANT

## Removal of bromine flame retardant by hot filtration

- The P2BC team developed a hot membrane filtration technology overcoming challenges of high viscosity materials in filtration



Viewing window into grey dissolved mixture of ABS + BFR



Permeate



Example of BFR fraction recovered from permeate



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## PLAST2bCLEANED DISSOLUTION BASED PROCESS SUMMARY



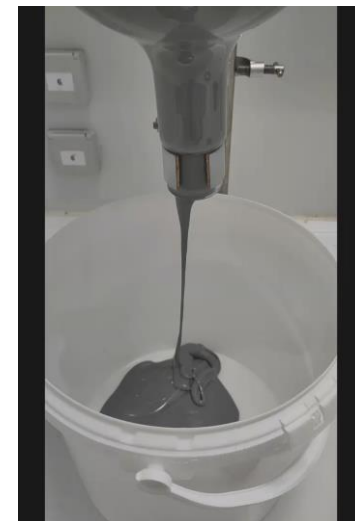
# KER I: RECOVERY OF ABS AND SOLVENT

## Drying of polymer solutions

- When removing solvent from the polymer viscosity increases significantly.
- Then solvent pockets form that enclose remaining solvent in the polymer.
  - Drying with oven, rotary evaporator is impossible.
  - Drying with vacuum extrusion is possible!
- The P2BC team tested spray drying as alternative with success



Spray dried rABS



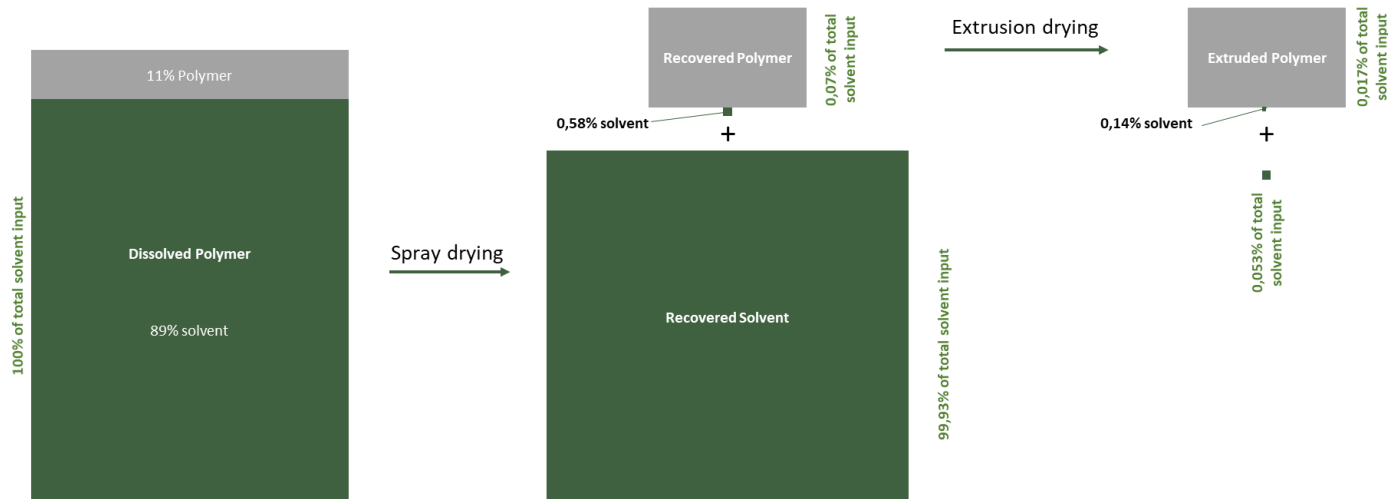
Tar-like viscosity of polymer-gel



# KER I: RECOVERY OF ABS AND SOLVENT

## Drying of dissolved polymer-mixtures by spray drying

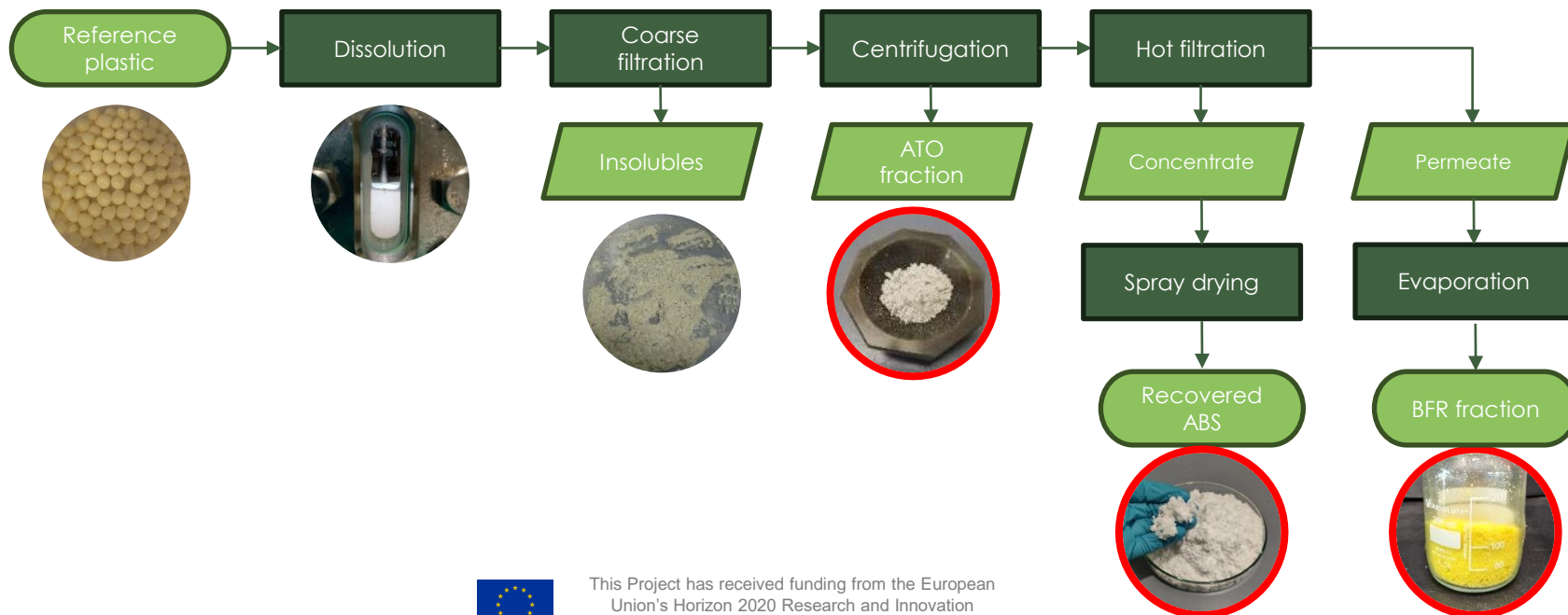
- Spray drying in one step removes >99,93% of solvent
- With added devolatilisation extrusion drying target specifications can be met (<1.000ppm)



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# PLAST2bCLEANED PROCESS SUMMARY

Lab scale testing on reference substrate; 3 fractions recovered

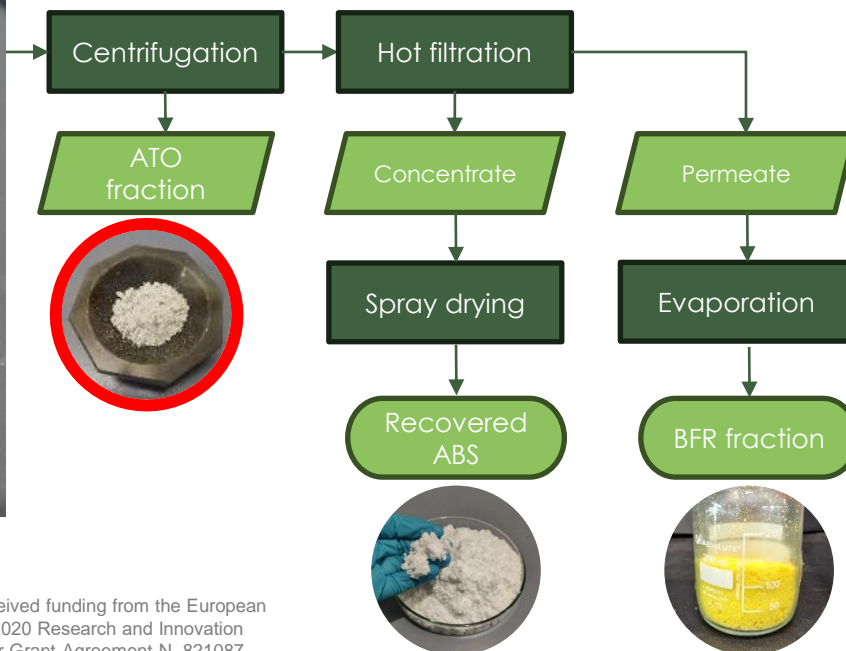
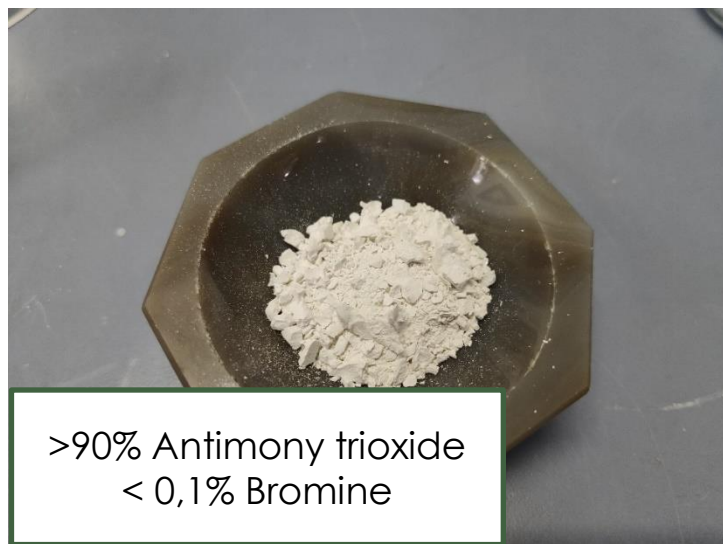


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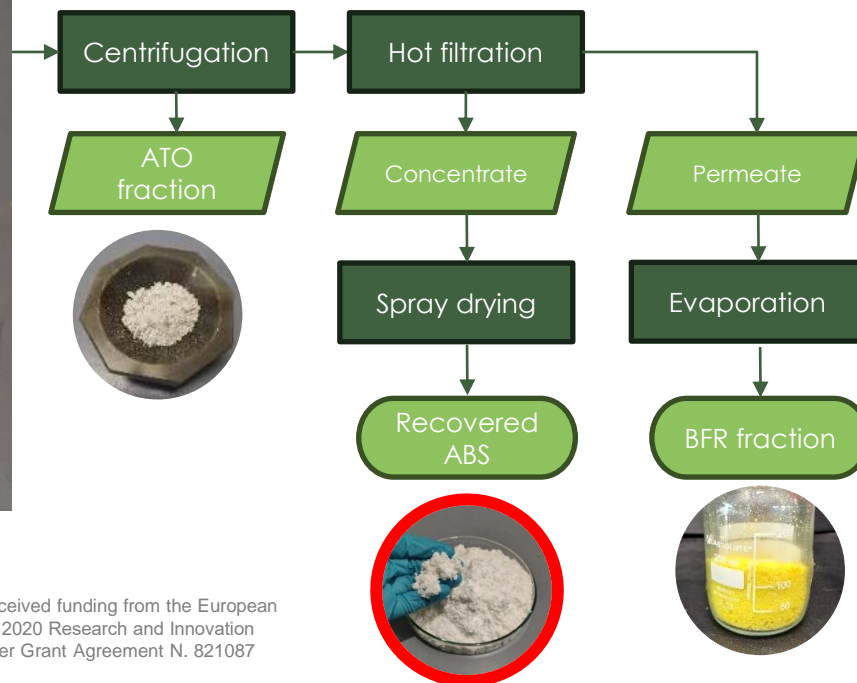
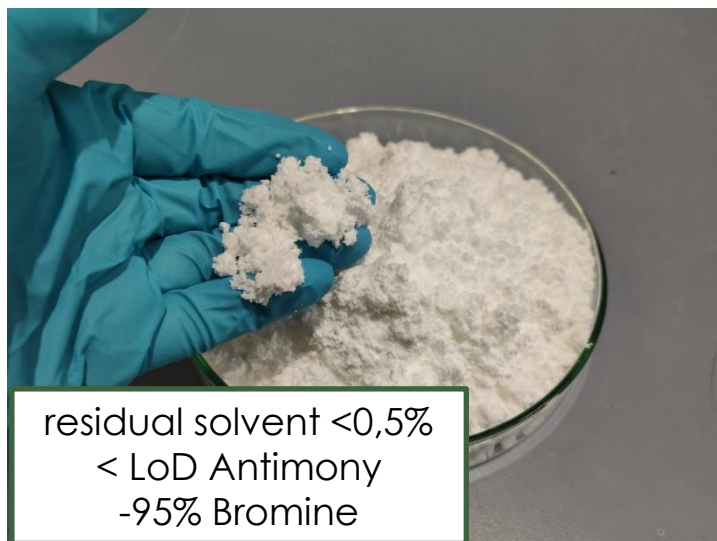
Lab scale testing on reference substrate



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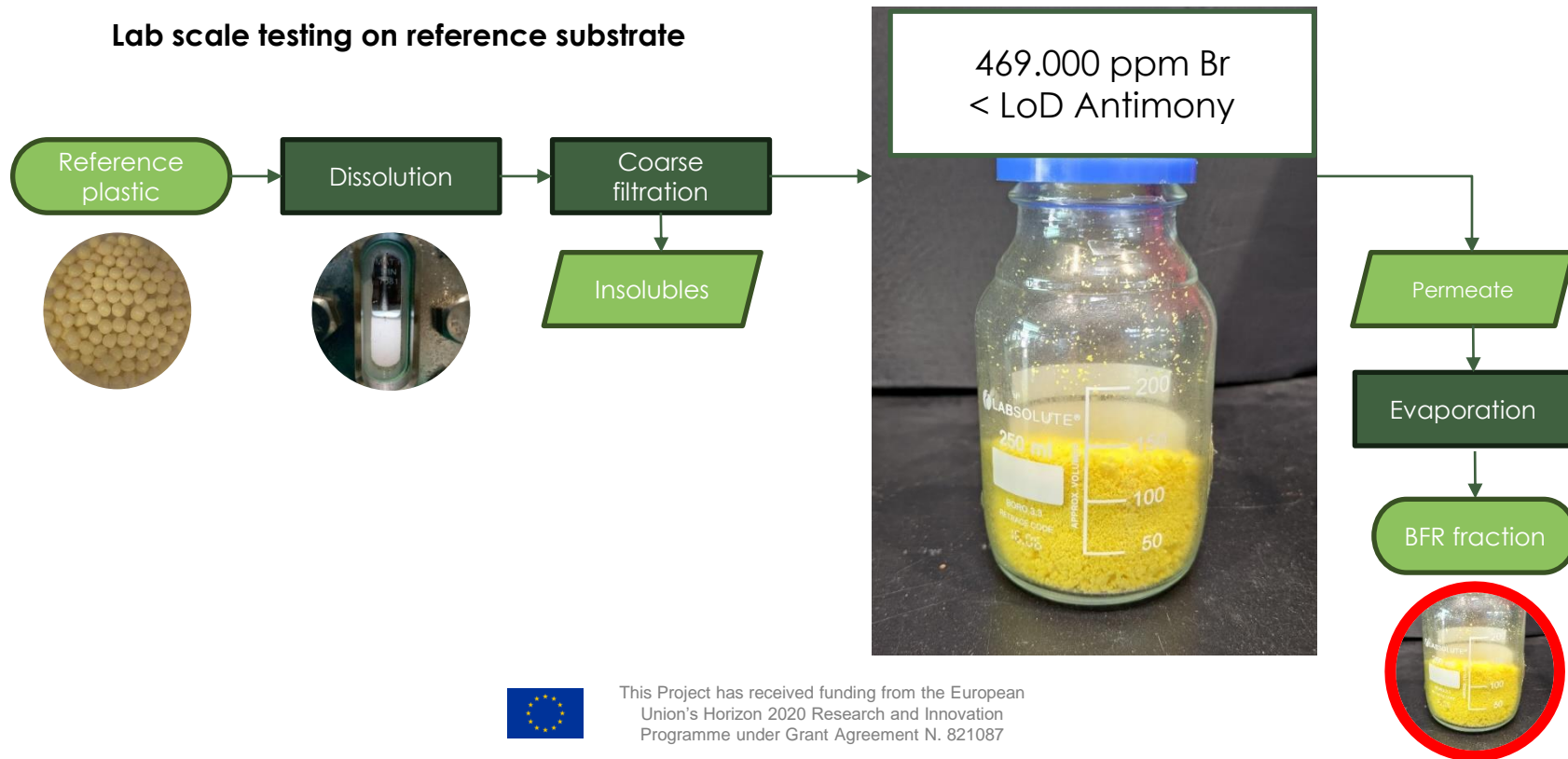
Lab scale testing on reference substrate



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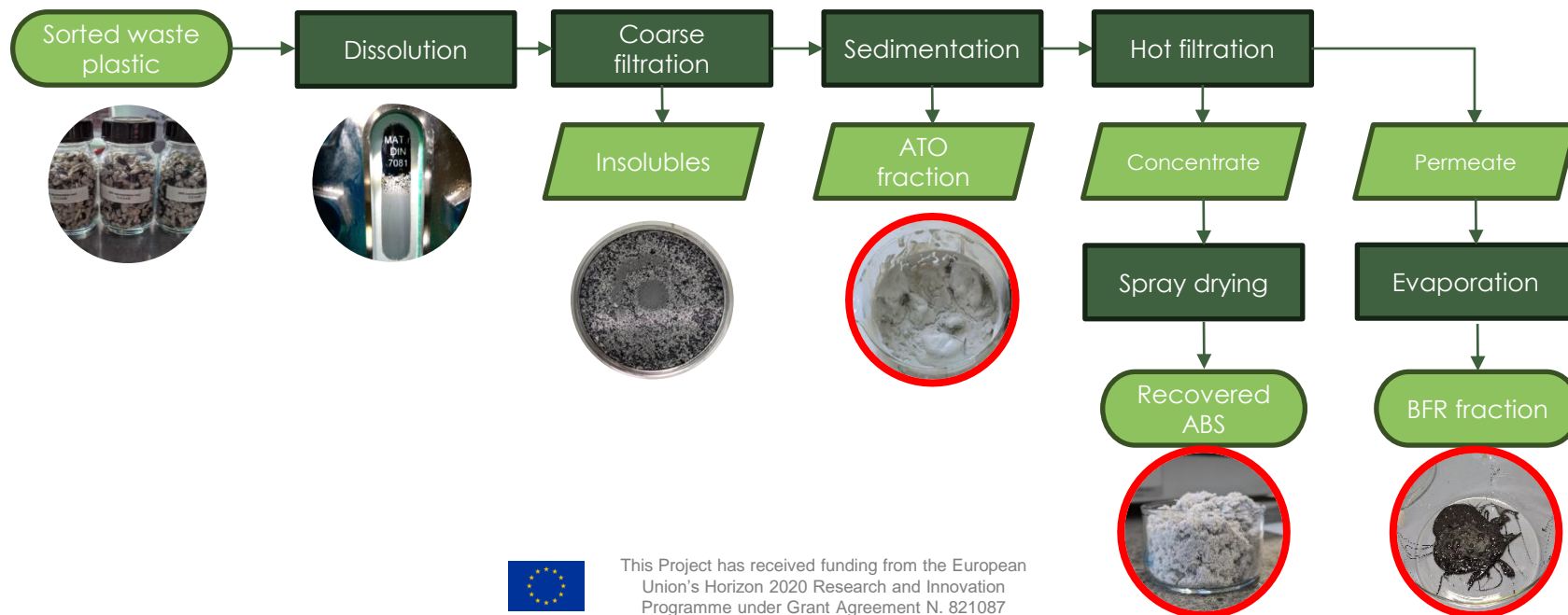
Lab scale testing on reference substrate



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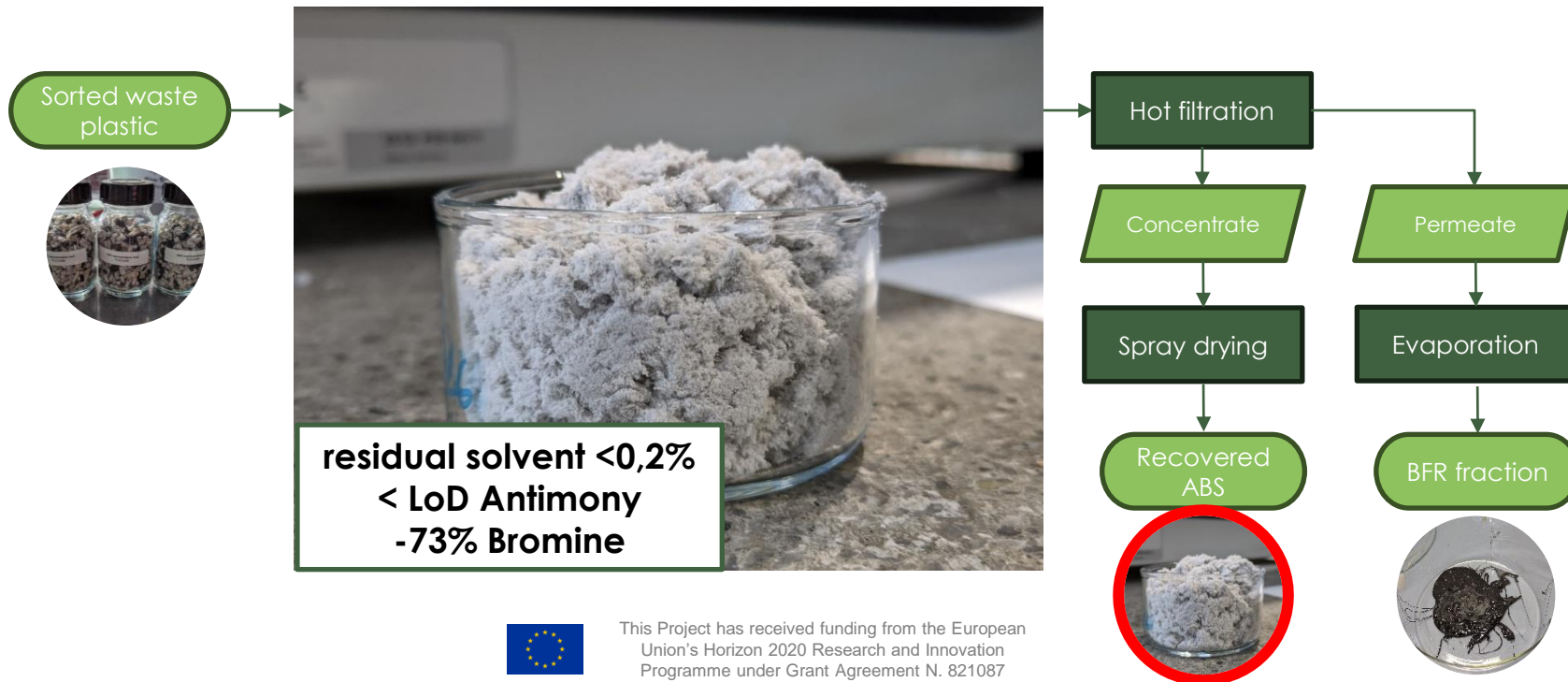
Lab scale testing on real waste; again 3 fractions recovered



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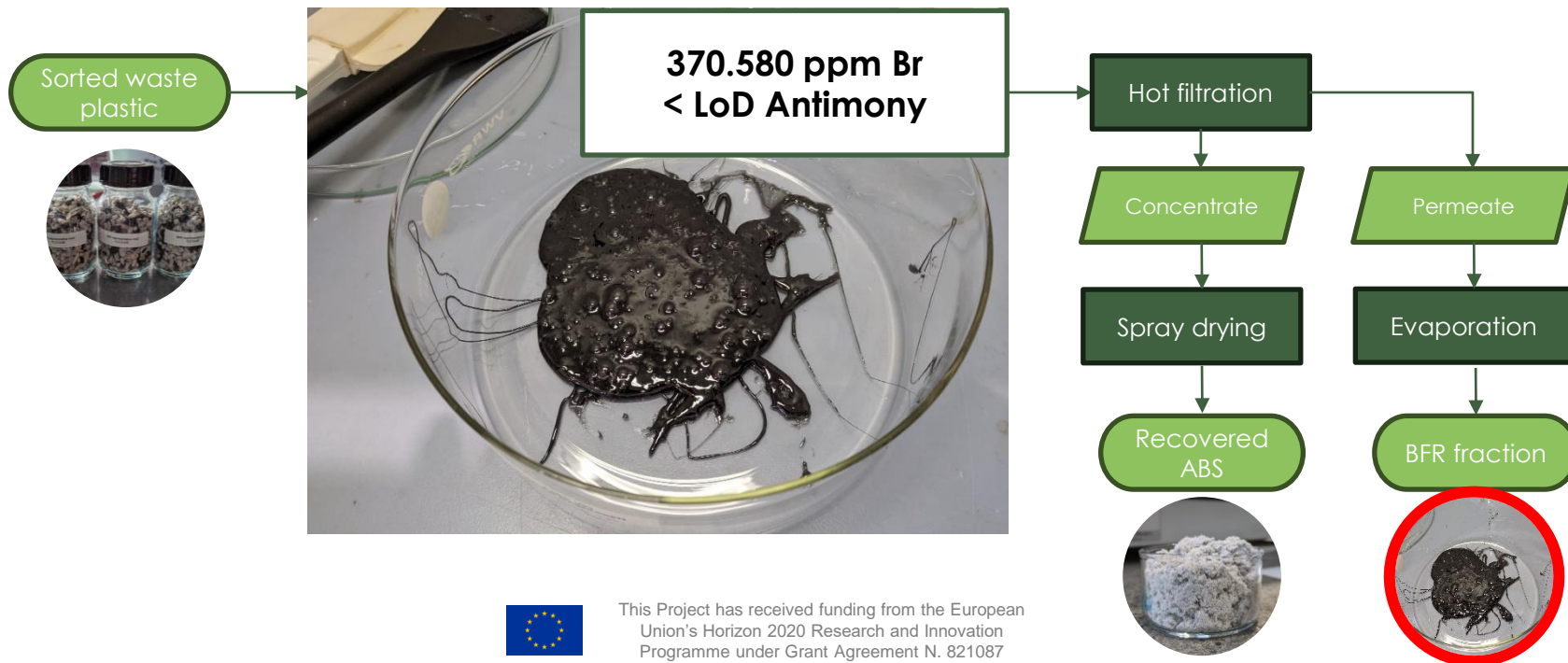
Lab scale testing on real waste



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# PLAST2bCLEANED PROCESS SUMMARY

Lab scale testing on real waste



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# SUMMARY

- The PLAST2bCLEANED project partners lead by TNO and Fraunhofer ICT developed a novel technology which includes

**KER I** → - a choice of solvent and successful dissolution method for waste plastics

**KER II**  - an antimony trioxide separation technology leading to high purity ATO on reference samples  
- a bromine flame retardant filtration technology removing between 95% (reference) and 73% (real waste) of bromine for recovered rABS

**KER I** → - a technique to remove 99.93% of solvent in one spray drying step leading to desirable dryness for reuse

- This technology was started at TRL 3 and scaled up to TRL 5 within the project to produce kg scale rABS

**KER IV:** open access TRL5 facility





## THANK FOR YOUR ATTENTION

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