DOE Bioenergy Technologies Office (BETO) 2023 Project Peer Review

- *Plastics Deconstruction and Redesign Panel*

Upcycling PET via the VolCat Process

April 4, 2023

Novel Methods for Deconstructing and Upcycling Plastics

BOTTLE FOA Topic Area 2

Award Number: DE-EE0009298

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VOLCAT: Transforming the Recycling of Dirty Plastics

Output (BHET)

PCR PET Feedstock
(dirty mixed waste)

Project Overview

- Context:
  - *Building on R&D by IBM Research, this project aims to optimize key unit operations, scale-up, and demonstrate an integrated process for a novel organocatalytic PET chemical recycling method known as VolCat.*
  - *NREL is dedicated to developing new technologies to deal with today's plastic wastes with new polymers and technologies for tomorrow's plastics.*
- Goals:
  - *Develop, optimize, and integrate the critical unit operations for VolCat based depolymerization and downstream processing (DSP) for realistic bottle flake and textile feedstocks.*
  - *Demonstrate full circularity by repreparing commodity fabric and bottles*
  - *Inform a robust TEA, LCA, energy flow and supply-chain models that can validate the promise of VolCat as a viable industrial technology for chemical recycling of waste PET bottles and textiles*

- Achieve ≥50% energy savings in closed-loop PET recycling and >40% GHG emissions reduction relative to virgin PET production with a ≥90% target for waste PET conversion.

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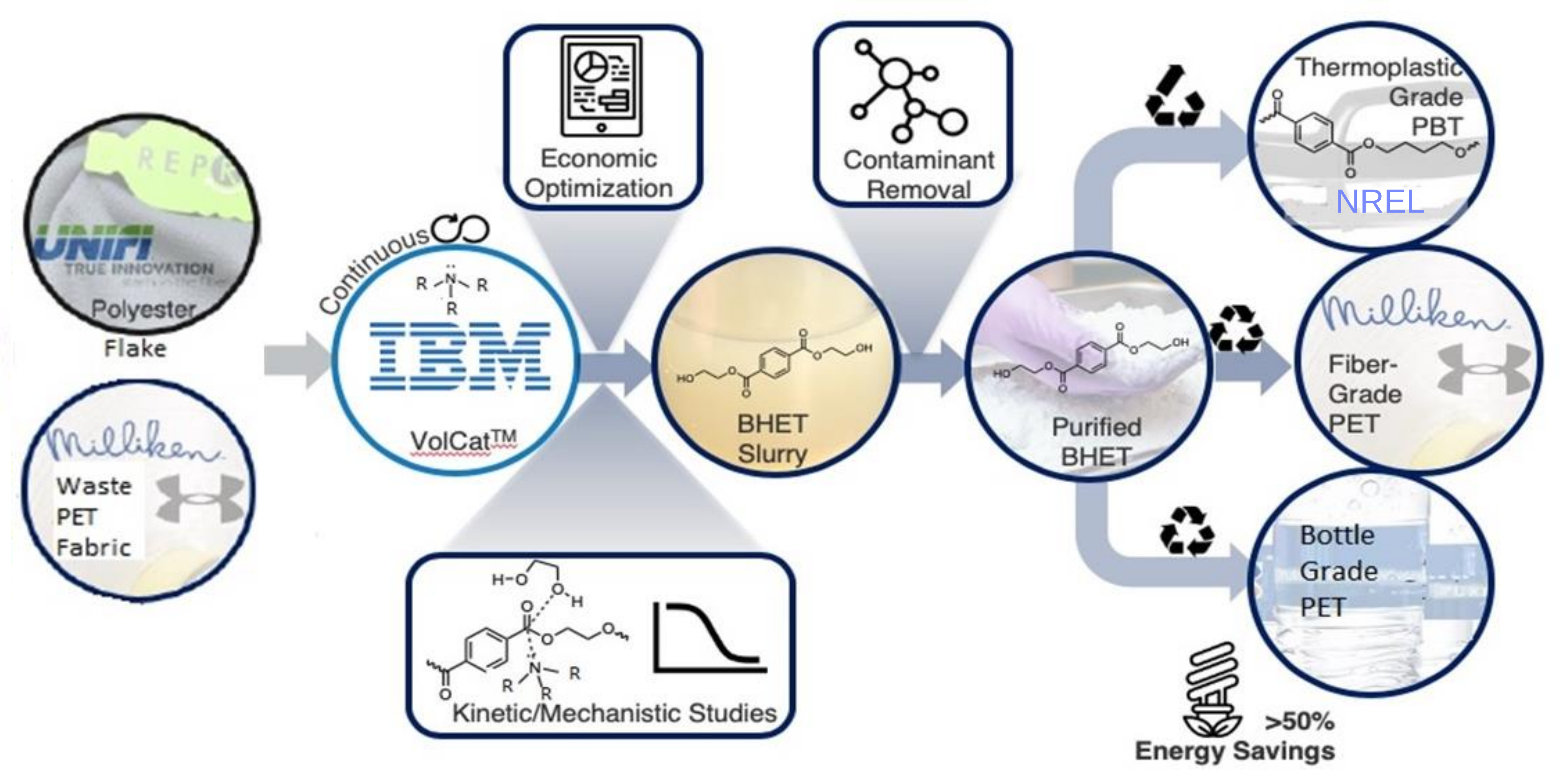
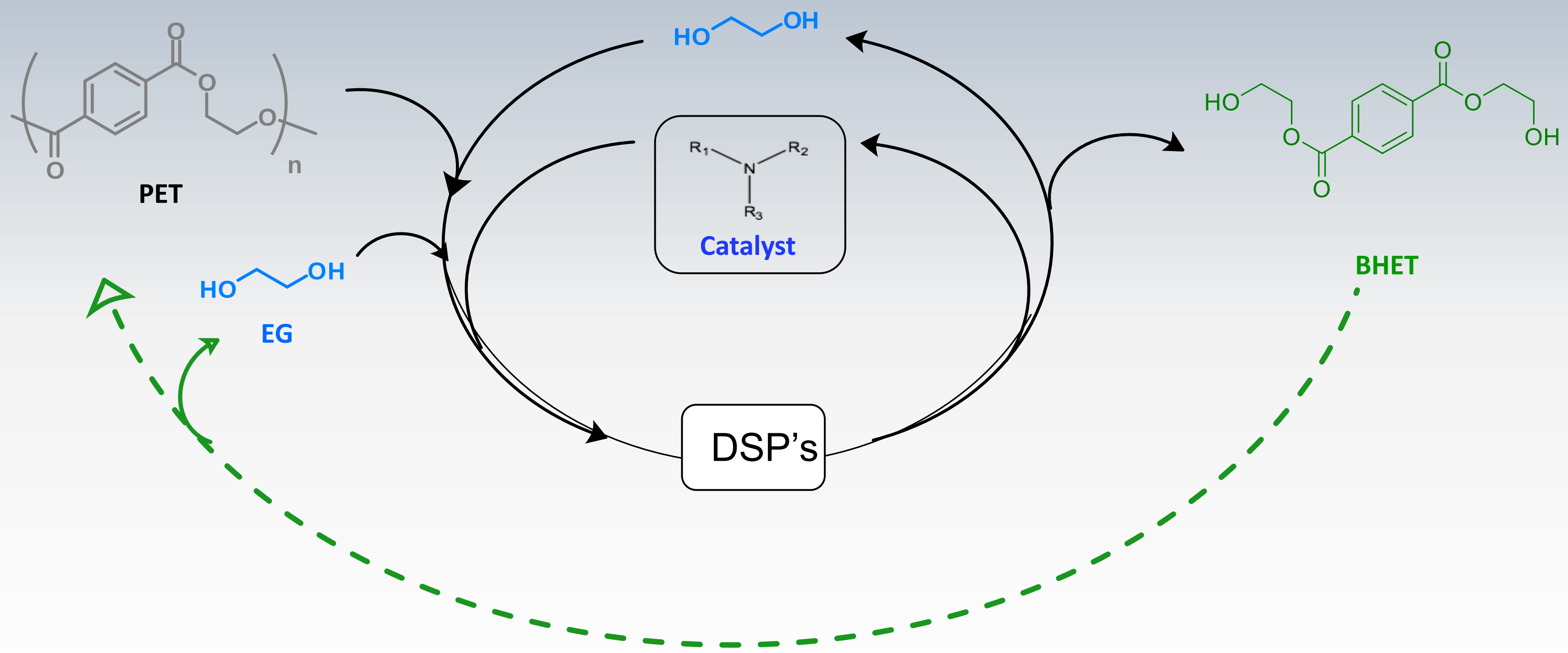
Approach

- *Demonstrate the baseline Volcat process for Verification*
  - *Showed >80% conversion of waste fabric and flake PET to BHET using "nominal" Volcat process*
- *Utilize a range of project partners to provide process inputs, scale the process, provide repolymerization capabilities and ability to reform and analyze commodity items*
- *Optimize the Volcat reaction parameters as well as the downstream process operations at the bench level utilizing realistic waste flake and fabric inputs.*
- *Feed results into the baseline TEA model to refine the results*
- *Perform Pilot scale demonstrations with waste fabric and flake inputs based on the optimized bench scale parameters*
- *Demonstrate Full recycling circularity using these reaction outputs to reprepared commodity-grade bottle and fabric*
- *Compare performance/quality of the re-formed commodities to existing materials (beverage bottle, clothing fabric)*
- *Prepare final TEA, LCA and GHG Emissions assessments*

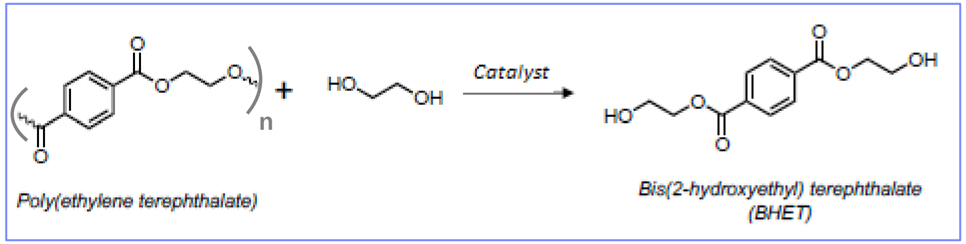
Project Flow Diagram



VolCat Closed-loop PET Recycling Scheme



- Closed-loop recycling process
- Closed loop depolymerization process
  - Catalyst recovered
  - EG recycled
- Only waste is that which arrived with PCR

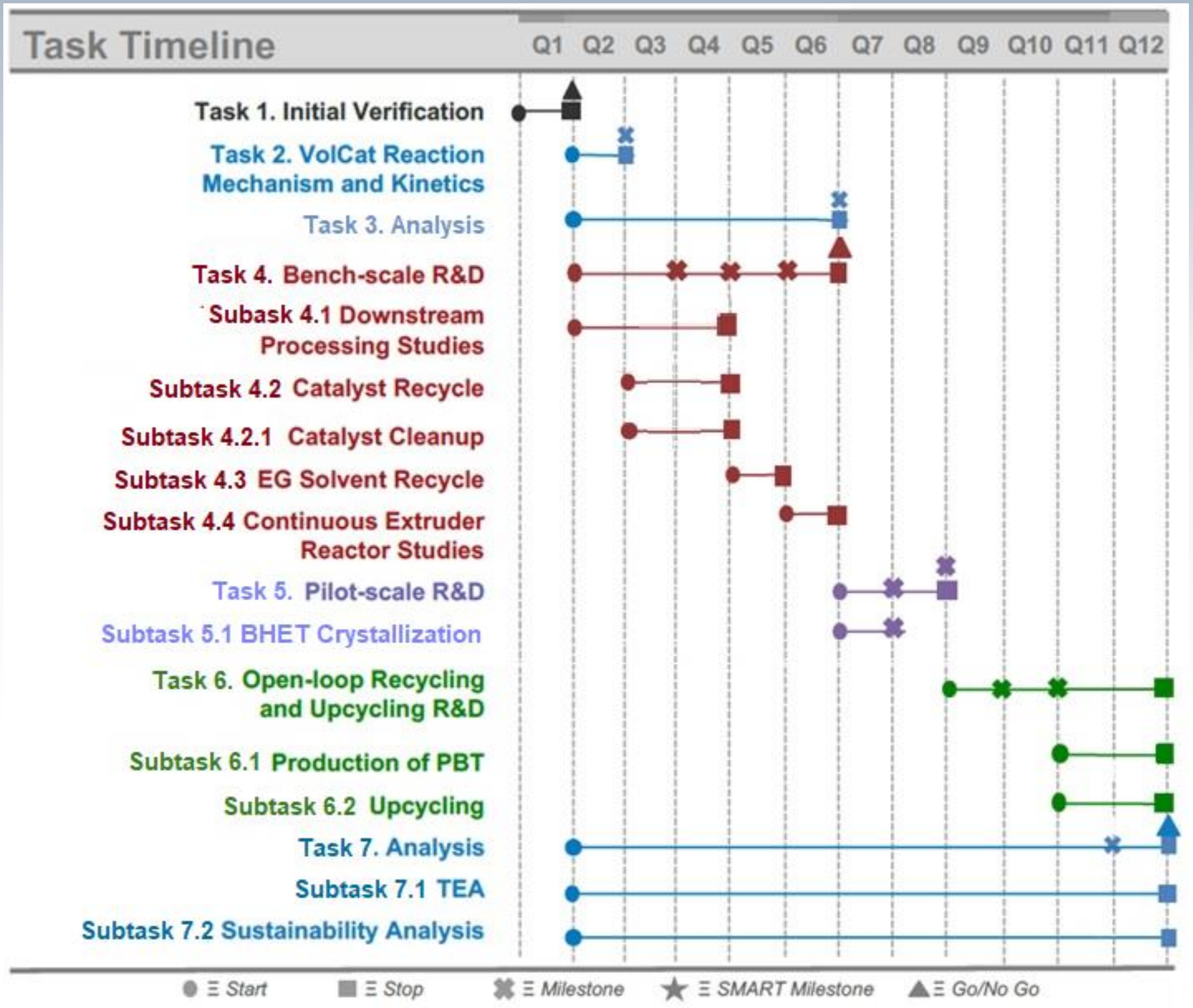


Approach

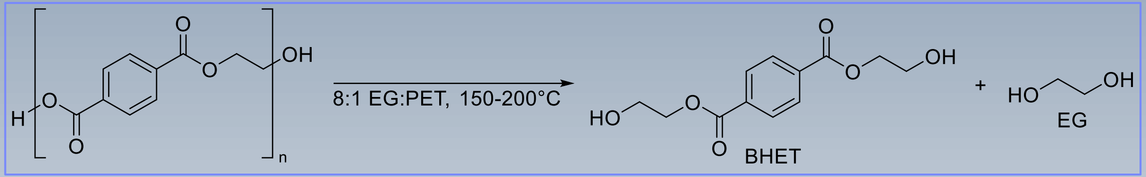
- ***Potential challenges***
  - *The desired optical quality (CIE color) for r-PET is well defined but the relationship of the BHET quality to good quality polymer outcome is not well known*
    - *What is the relationship of BHET monomer color to r-PET?*
    - *There are many potential impurities that may be unexpectedly detrimental to r-PET performance*
  - *Limited time in the project to “re-do” pilot scale reactions to regenerate commodities, these will likely be one-shot demonstrations*
  - *There is the possibility that an extrusion reactor may not be suitable to perform the Volcat reaction*
    - *Mitigation by exploring a Continuous Stirred Tank Reactor*

- *The successful completion of Go/No-Go Milestone 4.4 will greatly bolster confidence level for successful technology transfer to a pilot scale extrusion reactor*

Progress and Outcomes



Progress and Outcomes



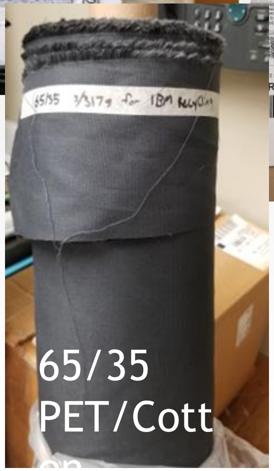
- ***Completed Task 2 Reaction Mechanism and Kinetics***
  - ***Milestone 2.1 met; Kinetics model for the Volcat reaction developed***
  - *Found a change of reaction pathway between reaction with and without use of organocatalyst*

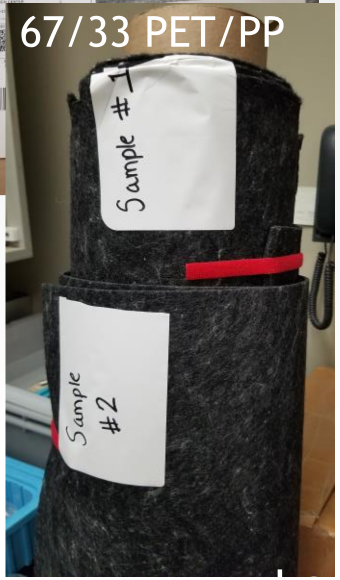
    The Arrhenius equation was used to find the activation energy of catalyzed end scission (21 kcal/mol), and the activation energy of uncatalyzed random scission (24 kcal/mol).
  - *No impact of agitation rate on depolymerization kinetics*
  - *A rather substantial effect of PET particle size.*
  - *A first order dependence on PET concentration was found, as well as a plateau of reaction rate at low catalyst loading*

- ***Task 3 Analysis (through 12/31/23)***
  - ***We will feed data from subsequent Tasks into the baseline model developed at NREL***

Auditioning of Proposed Inputs

- *Nominal process demonstrated on potential real-world waste inputs*





Milliken:

- *100% PET low grade non-woven and deadstock fabric*
- *Cotton and PP blended fabric*
- *High comonomer polyesters*

Under Armor:

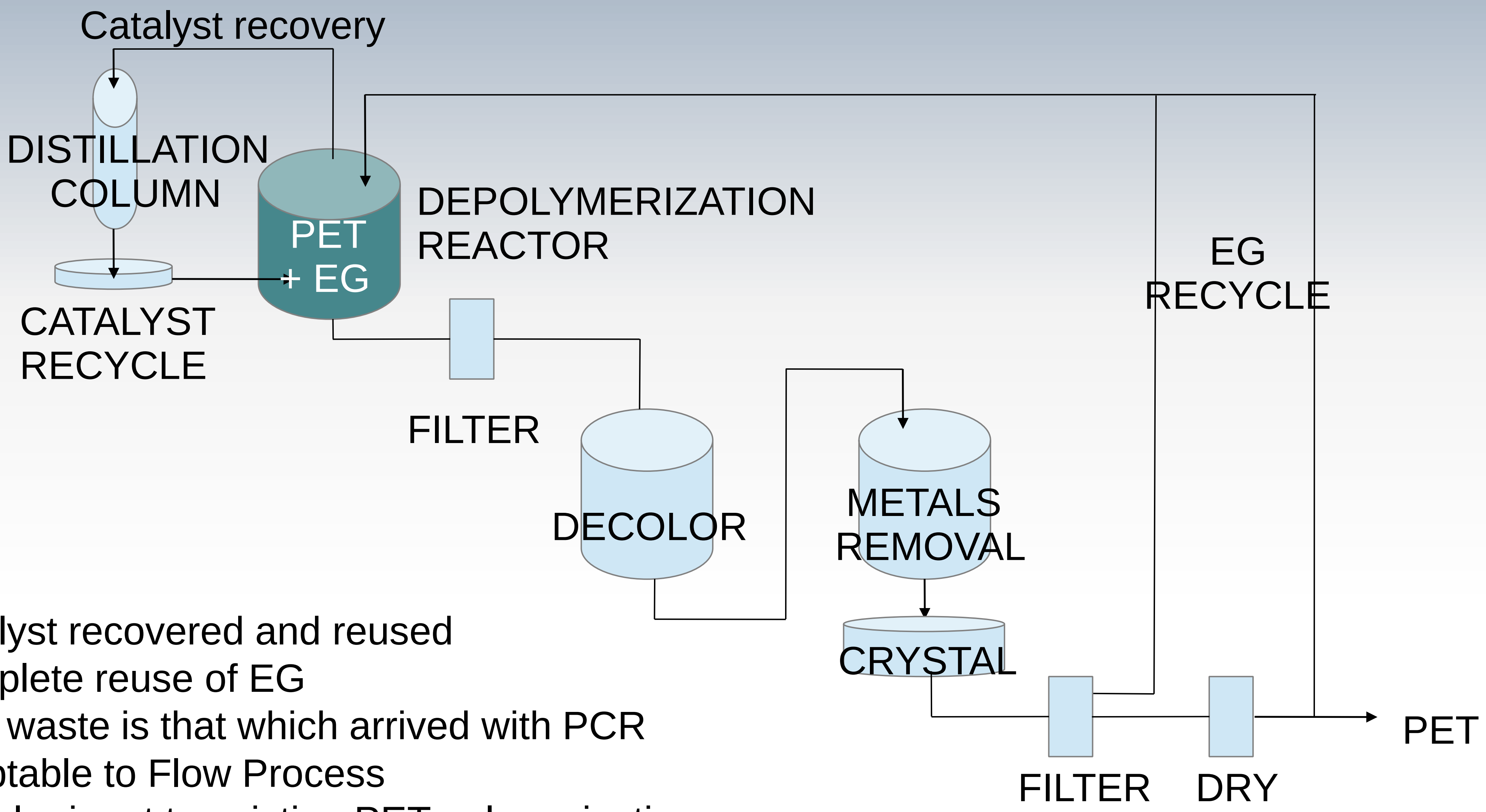
- *Industrial scrap fabric (mill waste)*

Unifi:

Various grades of bottle flake:

- *Low quality Sorter Rejects*
- *Unsortable Fines and unfit for use in mechanical recycling*

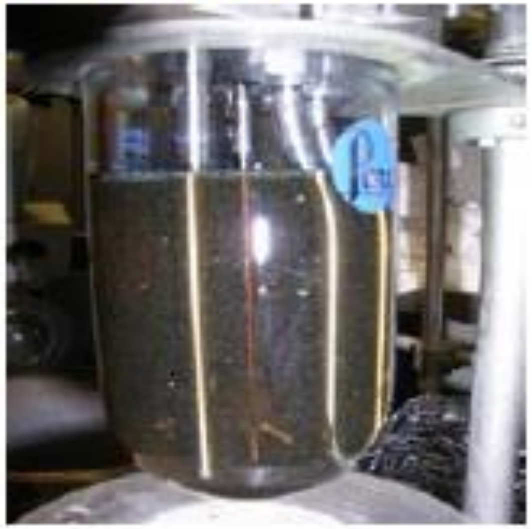
VOLCAT Process Flow – a closed loop process



- Catalyst recovered and reused
- Complete reuse of EG
- Only waste is that which arrived with PCR
- Adaptable to Flow Process
- Modular input to existing PET polymerization

Clean Colored Flake Input; 50 g Scale (exploratory)

"Pretty" to Start



"Pretty Ugly" Post-VOLCAT

Simple Filter Paper Filtration

Post Color Removal Treatment

Post Metals Removal

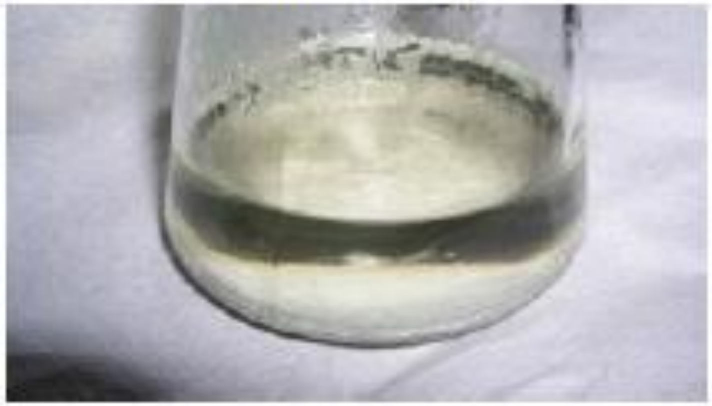
Dirty Mixed Flake Input; 50 g Scale (exploratory)

Start

Post-VOLCAT

Simple Filter Paper Filtration

Post Color Removal Treatment

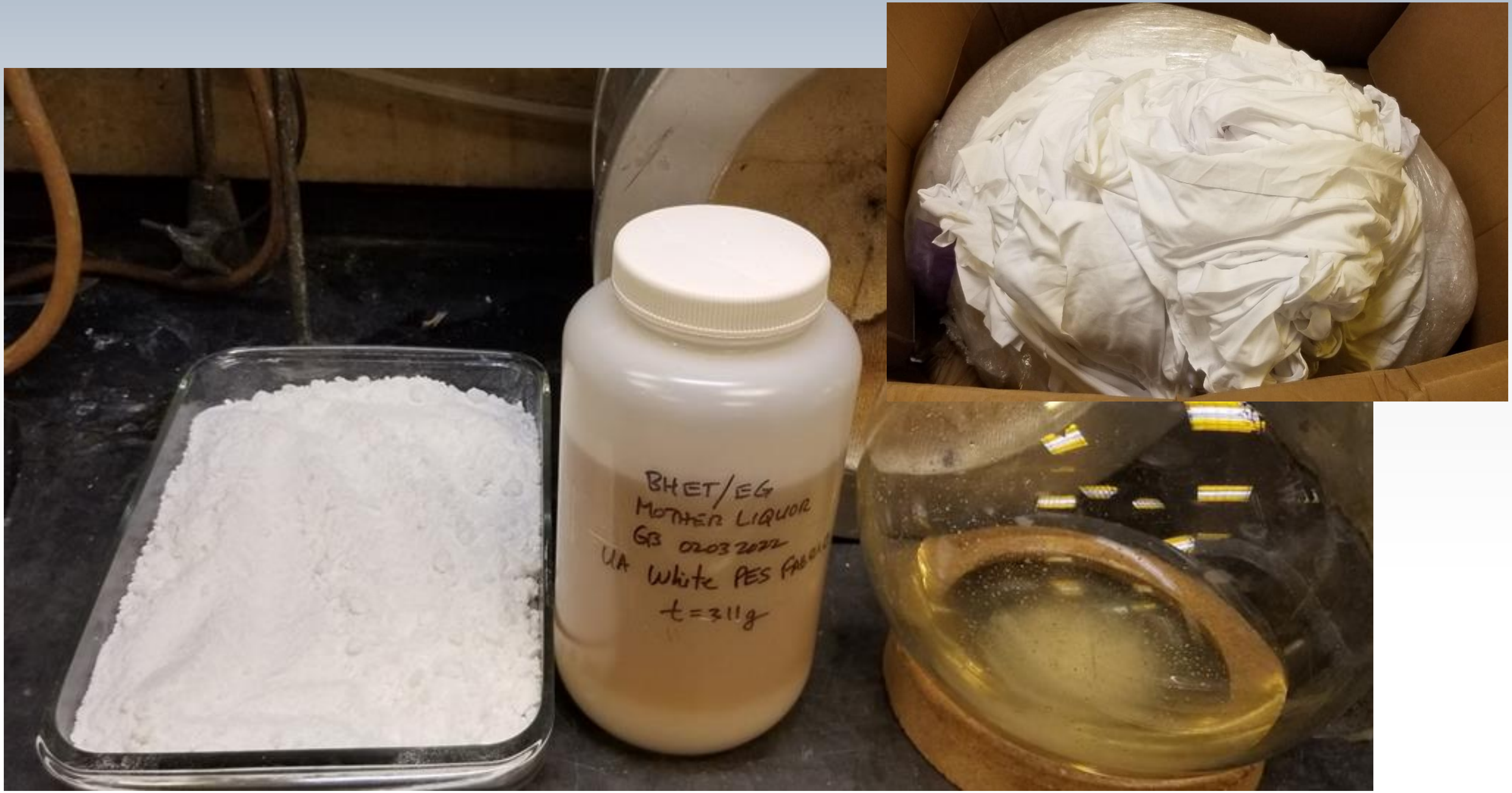


Post Metals Removal

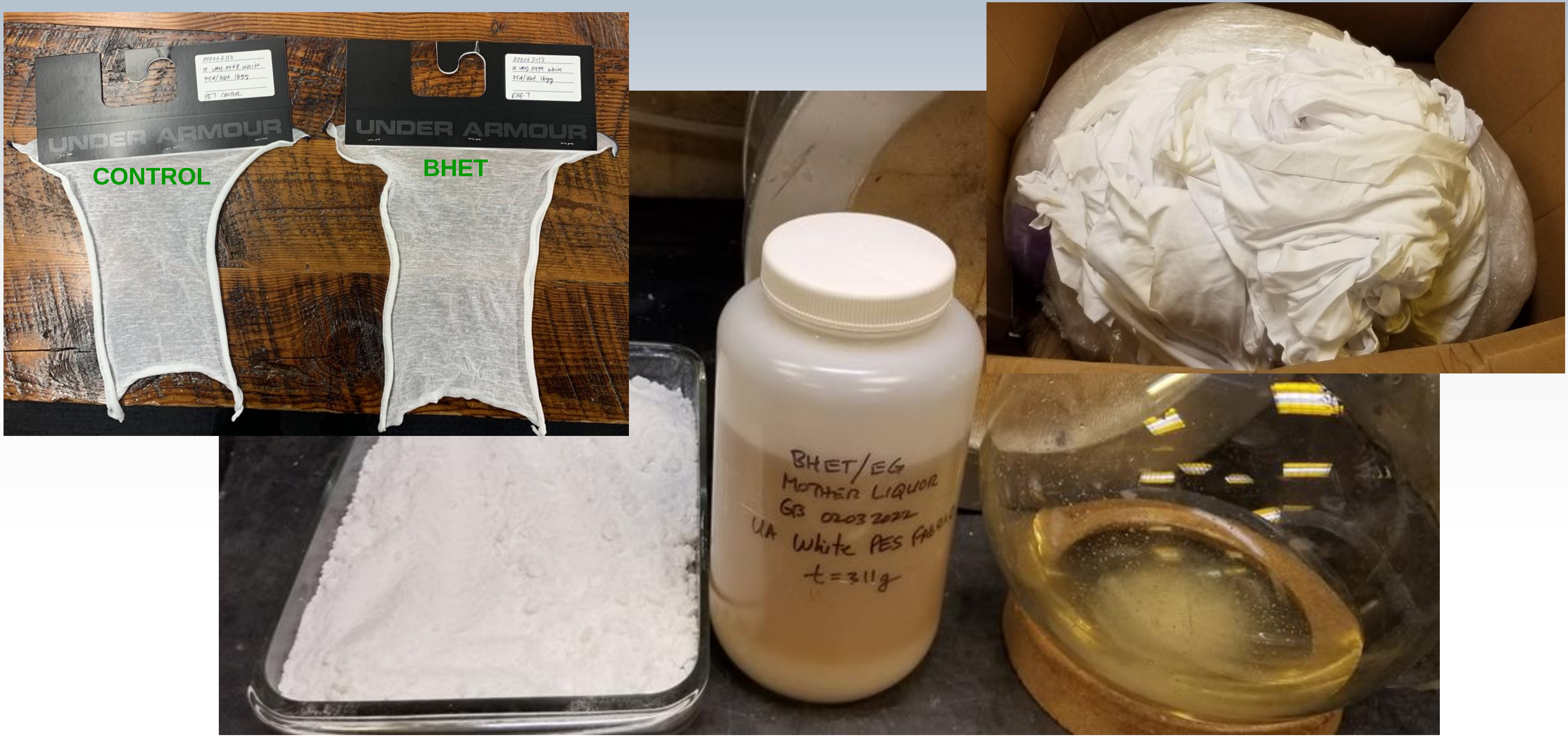
Input for demonstration – Flake from Unifi

BHET Sample Source (all Unifi)	L*	a*	b*	Comments
Fines, green	94.8	-2.7	2.3	Pre Pure
	97.6	-0.02	0.98	Post Pure
Mixed Color Rejects	91.2	0.1	4.9	Pre Pure
	98.1	-0.02	1.4	Post Pure
Color Rejects	94.1	-2.3	1.0	Pre Pure
	97.5	-0.1	0.96	Post Pure

Reaction Products from 100% Polyester fabric (1 kg Input)



De-risking Products from 100% Polyester fabric (1 kg Input)



Progress and Outcomes

- ***Task 4 Bench scale R&D (Until 12/31/23)***
  - ***Task 4.1 Downstream Optimization Studies (until 3/31/23)***

    Optimize decolor process

    Auditioned 7 process changes

    Found that this step can be accomplished using an order of magnitude less reagents than nominal process

    From ranked performance in color removal, found less expensive performers than nominal process

    Found process that was effective for both fabric and flake (different colorants used for each)

    Optimize metals removal process

    Just finishing this task with 4 process modifications

  - ***Expect to meet Milestone 4.1 labwork timeframe (3/31/23)***

    There will be a delay obtaining metals analysis from NREL

Progress and Outcomes

- ***Task 4 Bench scale R&D (Until 12.31/23)***
  - ***Task 4.2 Catalyst recycle (until 3/31/23)***

    Determine quantity and quality of catalyst recovery from reaction - In progress
  - ***Subtask 4.2.1 Catalyst Cleanup (until 3/31/23)***

    Found water content in some inputs distills over with catalyst

    Demonstrated easy phase separation to remove bulk of water

    Demonstrated further removal of water with dehydrating reagents – likely unnecessary
  - ***Expect to meet Milestone 4.2 (3/31/23)***
- ***Task 4.3 Solvent recycle (until 6/30/23)***
  - *Expect to meet Milestone 4.3; Recycle until steady state composition is obtained; solvent recovery >90%*
- ***Task 4.4 Bench scale Extrusion reactor studies (until 9/30/23)***
  - *Expect to meet **Go/No Go** Milestone 4.4; BHET yield of >80%*
  - *The parameters from this task will be transferred to Pilot Scale extruder (Task 5) to produce larger amounts of monomer*

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Progress and Outcomes - Summary

- *All auditioned inputs (with the exception of the highly colored cotton/polyester fabric) provided high-quality BHET using the nominal Volcat process.*
- *1kg Verification reaction using scrap fabric was the first time a fabric was scaled - results obtained are encouraging for scaleup using fabric inputs*
- *High quality r-PET and demonstration fabric obtained from verification product provides a baseline for quality to compare with post-optimization results*
- *Truncated decolorization process decreases cost by*
  - *Using less costly grade of reagent*
  - *Using much less than the nominal process (>10x)*
  - *Allows specifying smaller capacity equipment per product unit*
- *Decolorization process simplification due to unified reagent set for both fabric and flake waste inputs*
- *Easy removal of water impurity from catalyst recovered by the required recovery distillation*

Impact

- ***Currently mechanical recycling is limited to a fraction of the flake input available***
  - *Must be extremely clean and lack any color*
  - *Loss of IV on recycle requiring remediation*
  - *Tends to recycle impurities*
  - *No use of fabric inputs – missing ~60% of polyester market!*
- ***Volcat can utilize waste sources mechanical can't***
  - *Colored, unsorted, dirty, polymeric impurities*
  - *Can easily utilize fabric inputs*
  - *Removes known impurities (e.g. acetaldehyde, cyclic oligomers)*
- ***This project is a development activity slated for commercialization, not a research project***
  - *Focus will be to further the art by generation of patents over publications*
  - *Commercialization through licensing of the technology*
  - *JV partnership formed to commercialize this technology*

Project Team Acknowledgement

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  - *Greg Breyta*
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Quad Chart Overview

Timeline

- *Start: 10/1/2021*
- *End: 09/30/2024*

	FY22 Costed	Total Award
DOE Funding	*(10/01/2021 – 9/30/2022) $902,629*	*(negotiated total federal share) $3,547,090*
Project Cost Share	*$303,111*	*$1,051,465*

TRL at Project Start: 3
TRL at Project End: 6

Project Goal

Establish a technical baseline and developing process information necessary for scale-up. Inform robust TEA, LCA, and energy flow models that can validate the promise of VolCat as a viable industrial technology for chemical recycling of waste PET bottles and textiles.

End of Project Milestone

Demonstrate reduction of GHG emissions for bottle to bottle of greater than 40% vs virgin PET production and demonstrate of the full recycle of waste PET fiber and bottle back to commodity grade fabric and bottle products

Funding Mechanism

DE-FOA-0002245 **-** *Joint FY20 Bioenergy and Advanced Manufacturing FOA BOTTLE: Bio-Optimized Technologies to keep Thermoplastics out of Landfills and the Environment*

Project Partners

- NREL

Additional Slides

Publications, Patents, Presentations, Awards, and Commercialization

- N/A
- A JV has been formed to commercialize this technology