Polyester Digestion: VOLCAT

Summit on Realizing the Circular Carbon Economy

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IBM Almaden Research Center
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IBM Materials Innovation: Polymer Materials

IBM Technology

Grand Challenges

Lithography/Albany

QBIT Fabrication

Materials Innovation

AMD

COGNOZE

Computation/Machine Learning

Energy

Purification Materials

Catalysis/Green

Cognitive Therapeutics

Complex Separations

Antimicrobial Polymers

PET recycling

Next Gen Battery
Catalytic Polymer Recycling @ IBM

- IBM invests in polymer science research to help enable next generation computing
- Computational chemistry, materials simulation and accelerating materials discovery through AI/Machine Learning augment our experimental work
- Catalysts have been developed for the creation and breakdown of polymers
- We’ve developed a molecular sorter technology for r-PET (VOLCAT)
- We are interested in demanding applications moving these technologies to the next level through partnership

Jim Hedrick
Recycling of PET (r-PET): Challenges

**Mechanical Recycling**
- Sorting, washing (zero contamination tolerance)
- Only “non-colored” bottles
- High Temp Processing (T > 250°C)

**Chemical Recycling**
- Chemical Depolymerization
- Reaction Product (Monomer) Requires Purification for Polymerization
- Incorporated into Virgin PET Polymerization

COLOR/Low Quality  COST
IBM’s Organocatalytic depolymerization of PET

IBM’s Organocatalytic depolymerization of PET

Gen 1 Catalyst: High Activity, Difficult Recovery, $$

IBM’s PET catalytic depolymerization process (Gen 2 using DBU)

Heat to 190°C, add catalyst

Cool to Below 100°C Filter

Isolate BHET (yield approx 75%)

New PET

Solute, to be Recycled. Negligible Waste.

Clean
IBM’s PET catalytic depolymerization process (Gen 2 using DBU)

1. Heat to 190°C, add catalyst.
2. Cool to Below 100°C
3. Filter
4. Isolate BHET (yield approx 75%)
5. Clean
7. New PET

DBU Contamination/Loss!!

New PET
Computational prediction that the use of excess EG leads to a change in the mechanism allows for other types of amines to be used to catalyze PET depolymerization.
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Volcat Technology

- Fast/Selective Catalytic Process, low temperature, easy catalyst removal/recovery
- Key Attribute—**VOLCAT is a “Molecular Sorter”**
  - Minimize sorting, washing and rinsing of flakes
  - Reduce/eliminate color sorting
- Outstanding results with dirty clear and mixed/colored flake inputs
VOLCAT approach to recycled PET

Distinguishing Features:
- Volatile Catalyst is active and easily recovered;
- Low Grade Mixed PCR PET input can produce high grade r-PET
- Sorted Colored Flake can produce high grade r-PET

Economic Outlook:
- Lowest Cost Feedstocks and inexpensive catalyst
- All chemicals used/recovered (no waste);
- Low temperature/fast process

Other Advantages:
- Potential to reduce / eliminate sorting
- Potential to reduce / eliminate washing and rinsing
VOLCAT Process Flow Today – a closed loop (as batch) process

- Complete recovery of catalyst
- Complete reuse of EG
- Only waste is that which arrived with PCR
- Adaptable to Flow Process
- Modular input to existing PET polymerization
Robust process tolerant of high degrees of contamination

Dirty Mixed Flake

Unsorted, uncleaned flake; cheap input
-A considerable amount of dirt is present (~4%)

Clean Mixed Flake

All colored Flake from post-sorting/cleaning
Cheapest input – little commercial use

PET/Nylon & PET/PVC flake sample

- PET flake with 3 wt% Nylon
- PET flake with 3 & 0.1 wt% PVC (NURRC + Aldrich)

Worst of the worst. “Curbside pickup”
- Large amount (~8%) of “dirt” is present; cheapest input
VolCat Process with Clean Color Flake

From reactor

The VOLCAT product filtered to remove this small amount of “blue stuff” (some flake)

To give this turbid solution TO WHICH carbon was added

Filtration of carbon (left) to IX Treatment flask

Solution after post-IX filtration. Faintly blue, left to crystallize

To produce this! – Filtration next.
VolCat Processed Colored Flake (5 kg)

BHET Product (polymerization grade)

All inputs provide similar results
Filtration Step Comparison

<table>
<thead>
<tr>
<th></th>
<th>Clean Colored</th>
<th>Dirty Mixed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight (g)</td>
<td>0.05</td>
<td>2.1</td>
</tr>
<tr>
<td>Percentage (%)</td>
<td>0.5%</td>
<td>4.2%</td>
</tr>
</tbody>
</table>

Curbside Dirty Mixed: 3.6g (7.2%)
BHET polymerization to PET*: Polymer Characterization

Any/all feeds can be “VOLCAT-ed” to produce bottle grade BHET (50%)

<table>
<thead>
<tr>
<th>BHET Monomer Source</th>
<th>Transparency (L*)</th>
<th>Color (b*)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Virgin PET (from TPA/EG)</td>
<td>93</td>
<td>2.3</td>
</tr>
<tr>
<td>Virgin PET; Glycolysis (re-polymerized)</td>
<td>87</td>
<td>8.0</td>
</tr>
<tr>
<td>VOLCAT BHET (clean, clear flake)</td>
<td>93</td>
<td>2.1</td>
</tr>
<tr>
<td>VOLCAT BHET (dirty clear)</td>
<td>92</td>
<td>5.9</td>
</tr>
<tr>
<td>VOLCAT BHET (colored flake)</td>
<td>92</td>
<td>5.6</td>
</tr>
<tr>
<td>VOLCAT BHET (curbside, dirty)</td>
<td>92</td>
<td>4.5</td>
</tr>
</tbody>
</table>

* Performed by DAK Americas, an Alpek Polyester Business
PET Chemical Recycling via VOLCAT

Feedstock Insensitivity

Process Scalability?  
Process Economics?  
New Materials?

BHET Monomer

PET Waste

High Monomer Purity

PET

High Polymer Purity
PET Chemical Upcycling via VOLCAT

Feedstock Insensitivity

PET Waste

PET-derived AMIDE Monomers

BHET Monomer

High Monomer Purity

PET

High Polymer Purity

Process Scalability?

Process Economics?

New Materials?
Catalytic Polymer Recycling @ IBM: Other Processes

- PHT Thermosets: Depolymerization at low pH
- Conversion of Poly(carbonate) to Poly(ether sulfone)

Jeannette Garcia

poly(BPA carbonate)  
\[ M_n \approx 12,000 \text{ Da} \]

poly(aryl ether sulfone)  
\[ M_n \approx 11,000 \text{ Da} \]