Polypropylene Process Overview

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TUTORIAL
FEB 26, 2017

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Public

Dr. C.P. Cheng
BU Catalyst
Propylene and Aromatics
26.02.2017

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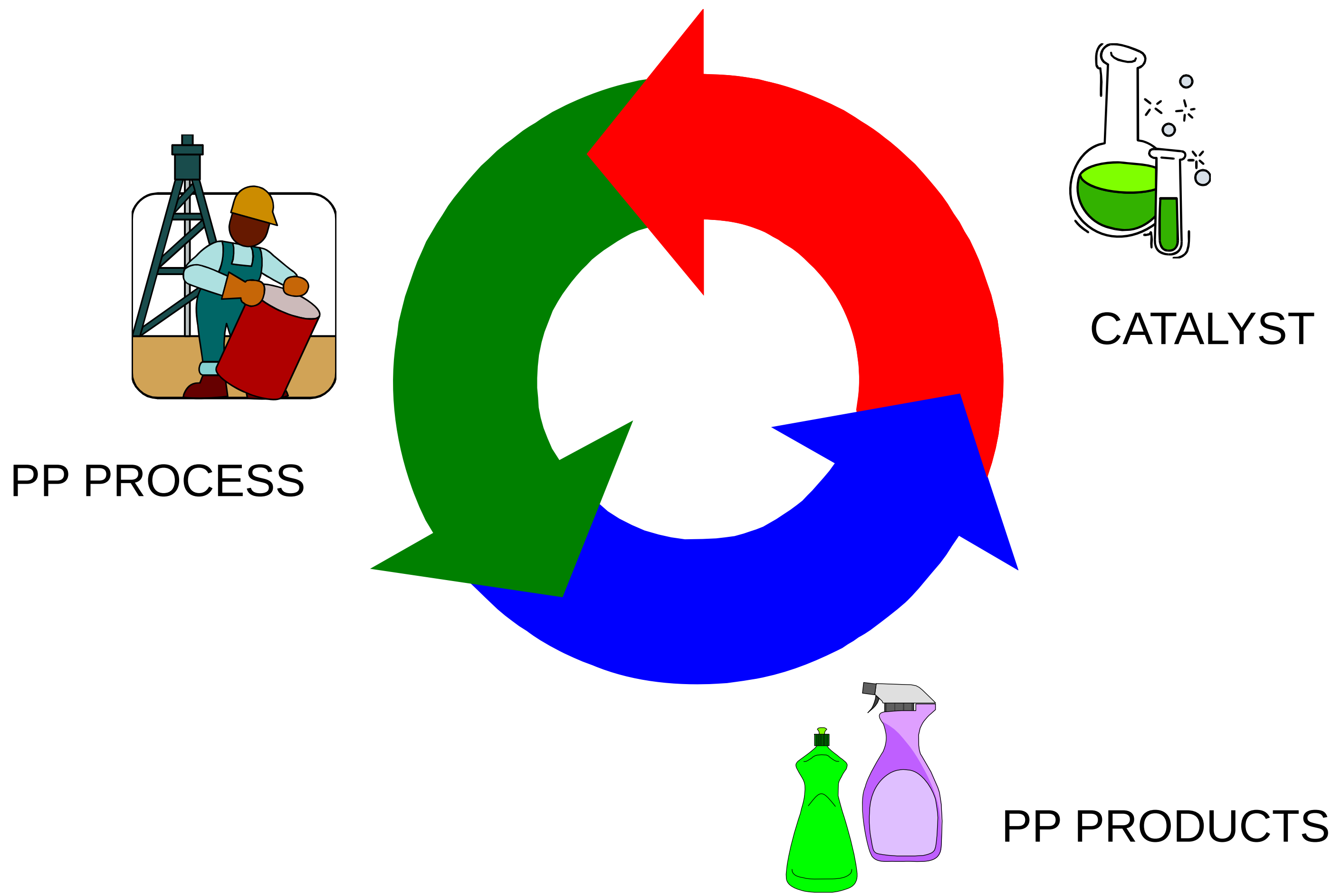

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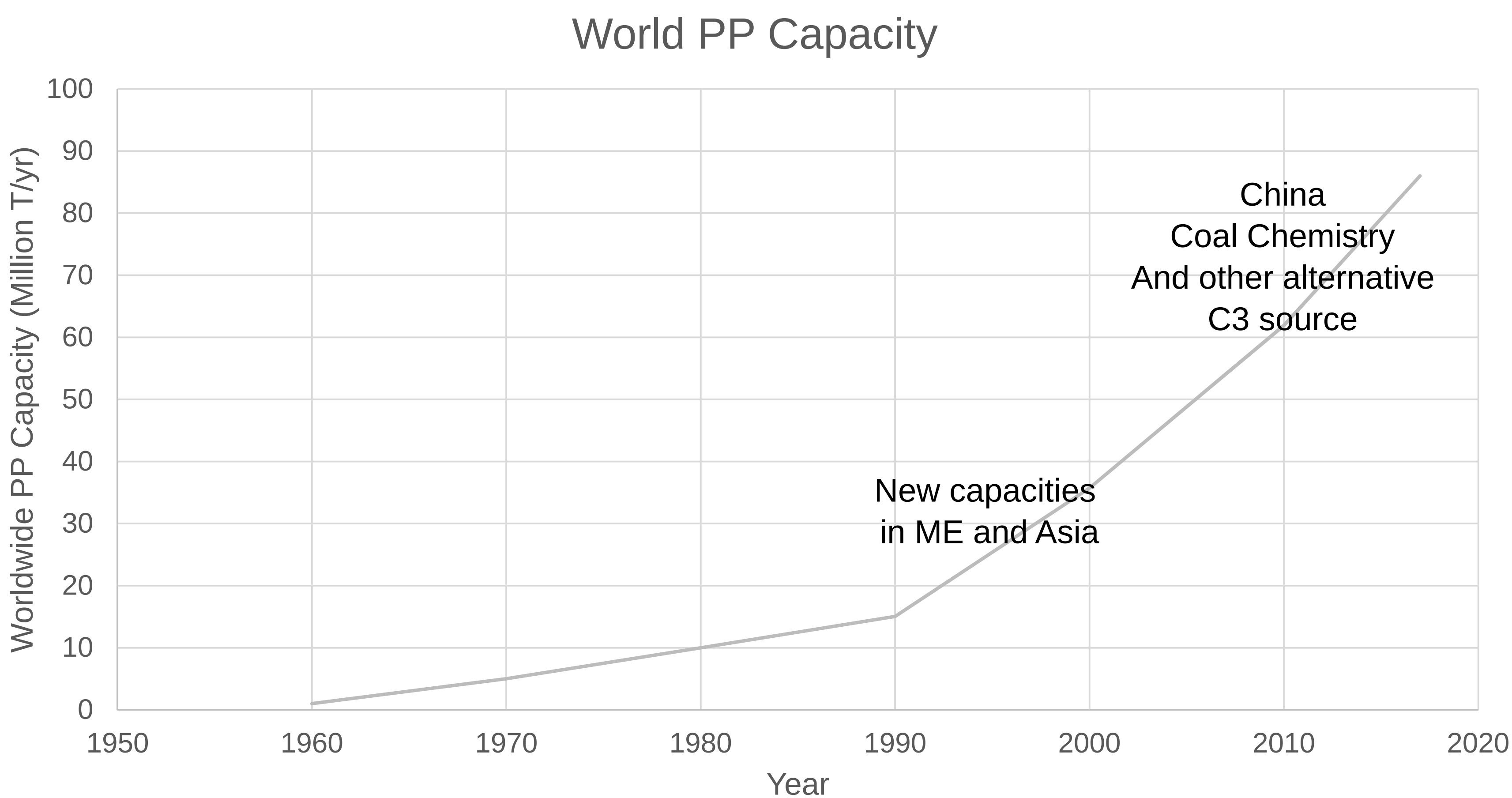
Worldwide PP Installed Capacity



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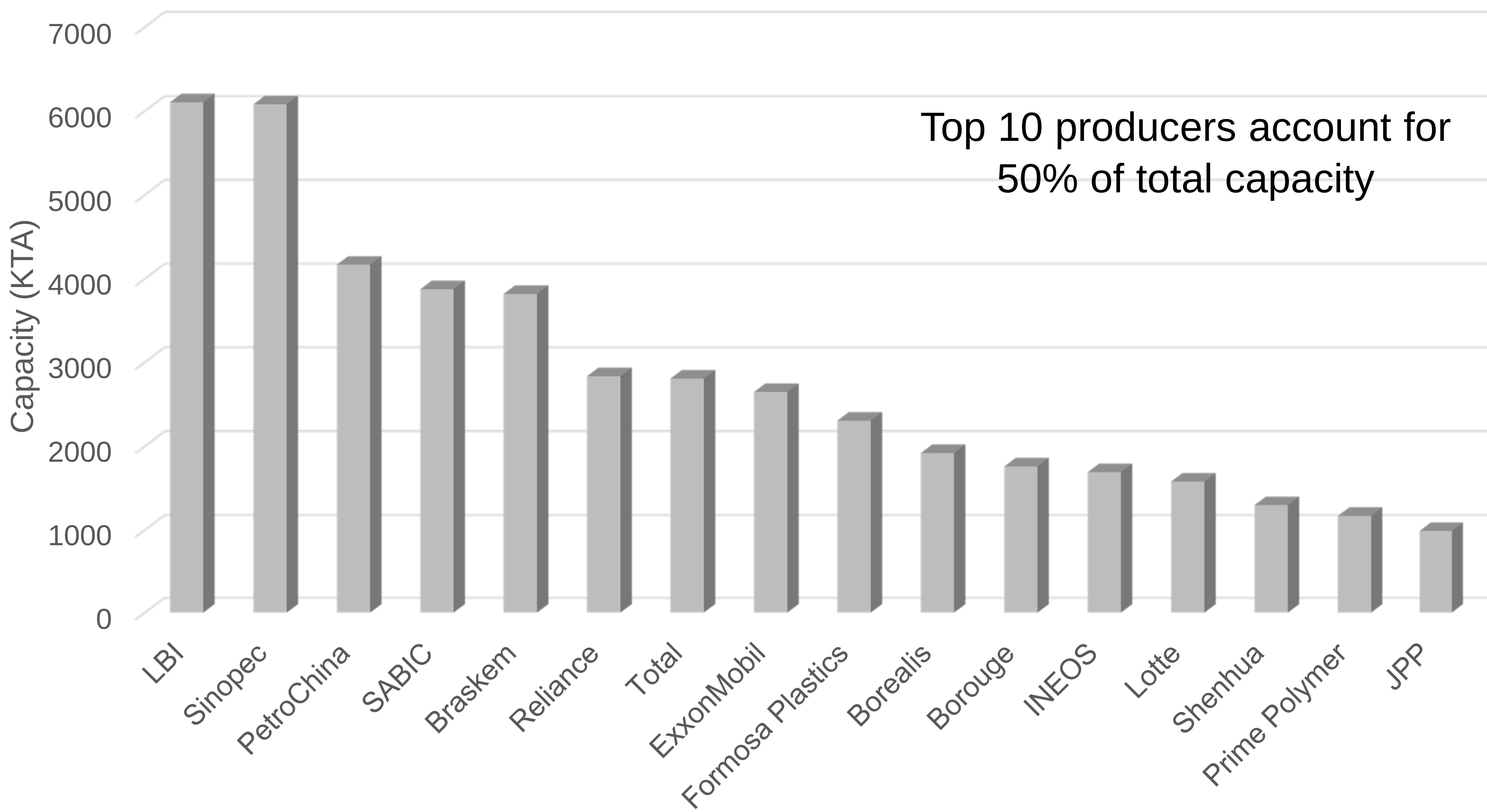
Top PP Producers



Source: Townsend Solutions

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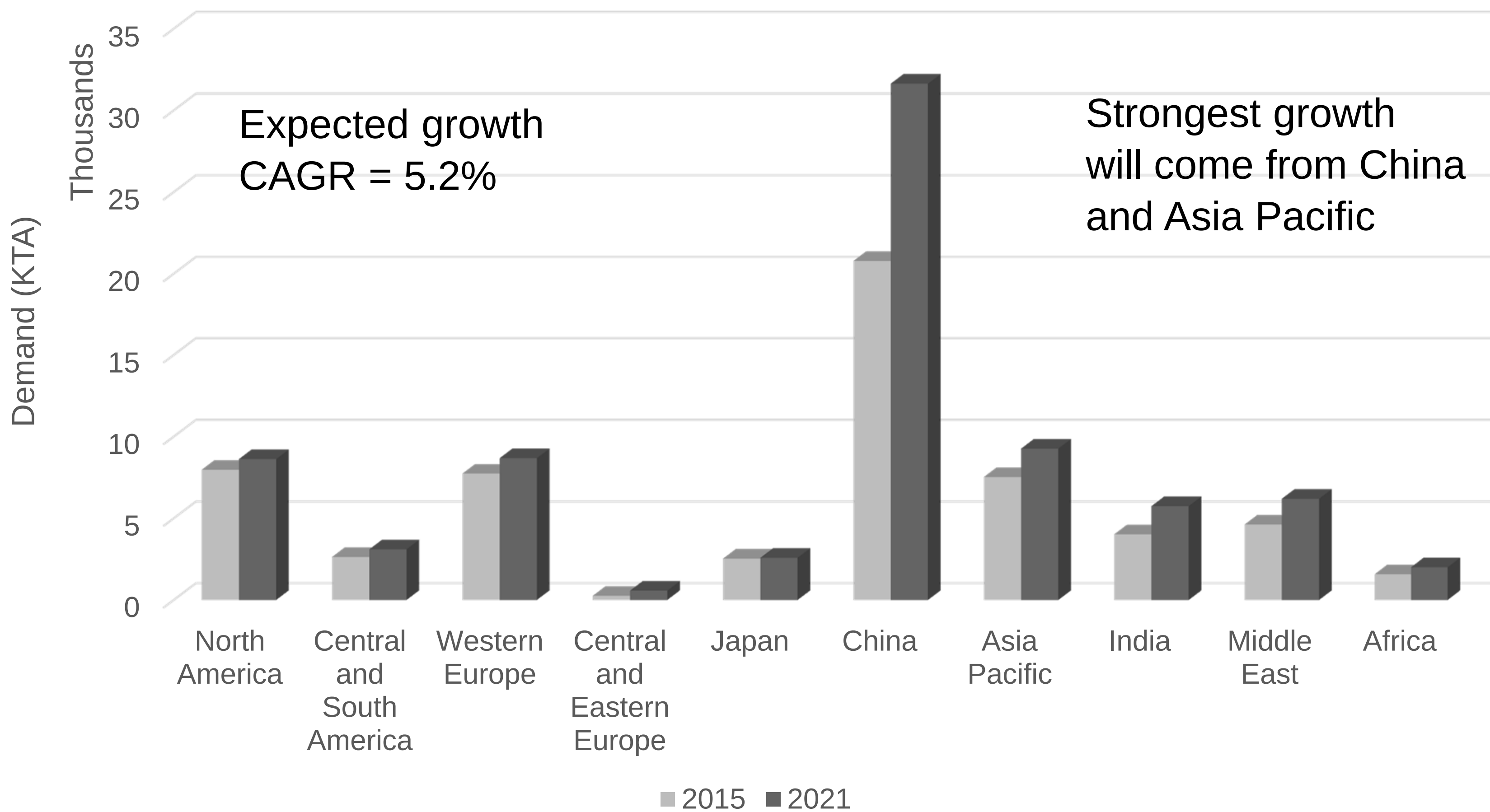
Worldwide PP Demand by Region



Source: Townsend Solutions

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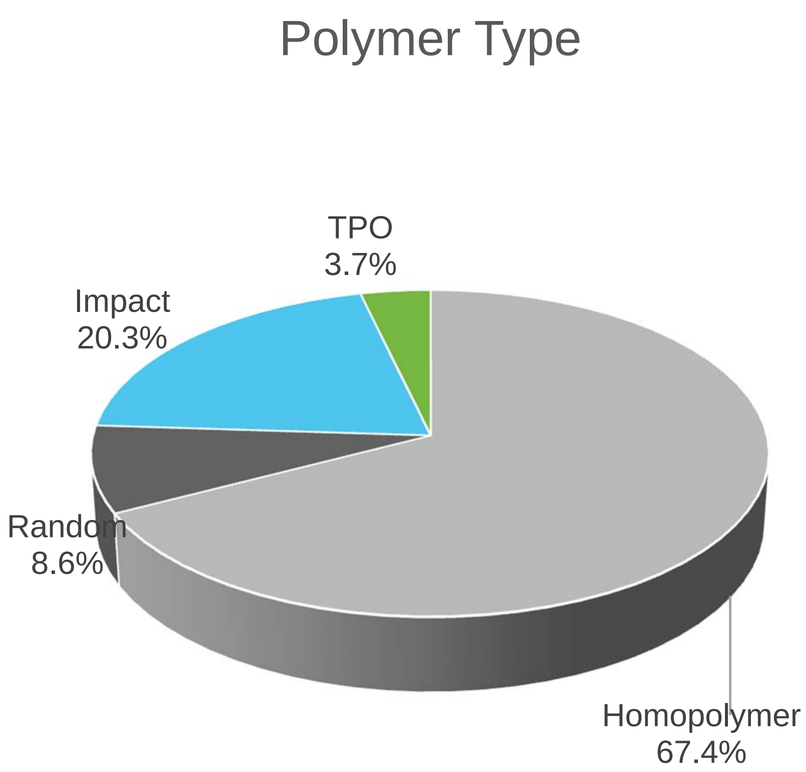
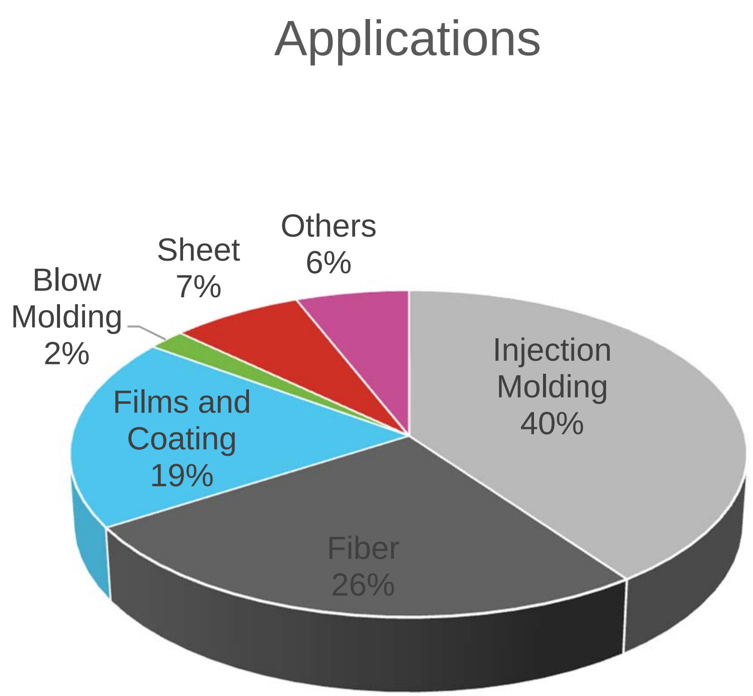
PP Demand by Applications





Source: Townsend Solutions

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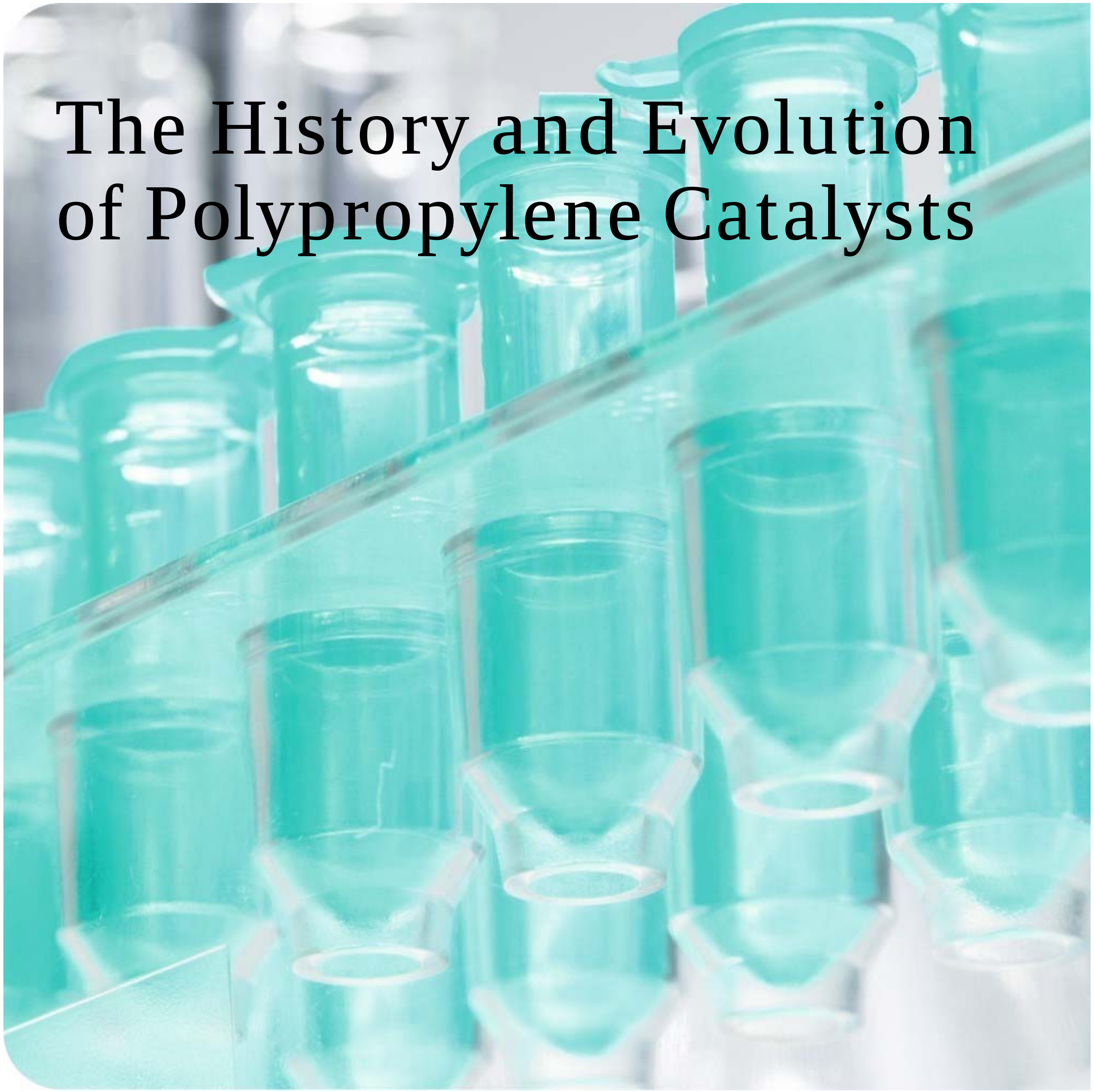

The History and Evolution of Polypropylene Catalysts

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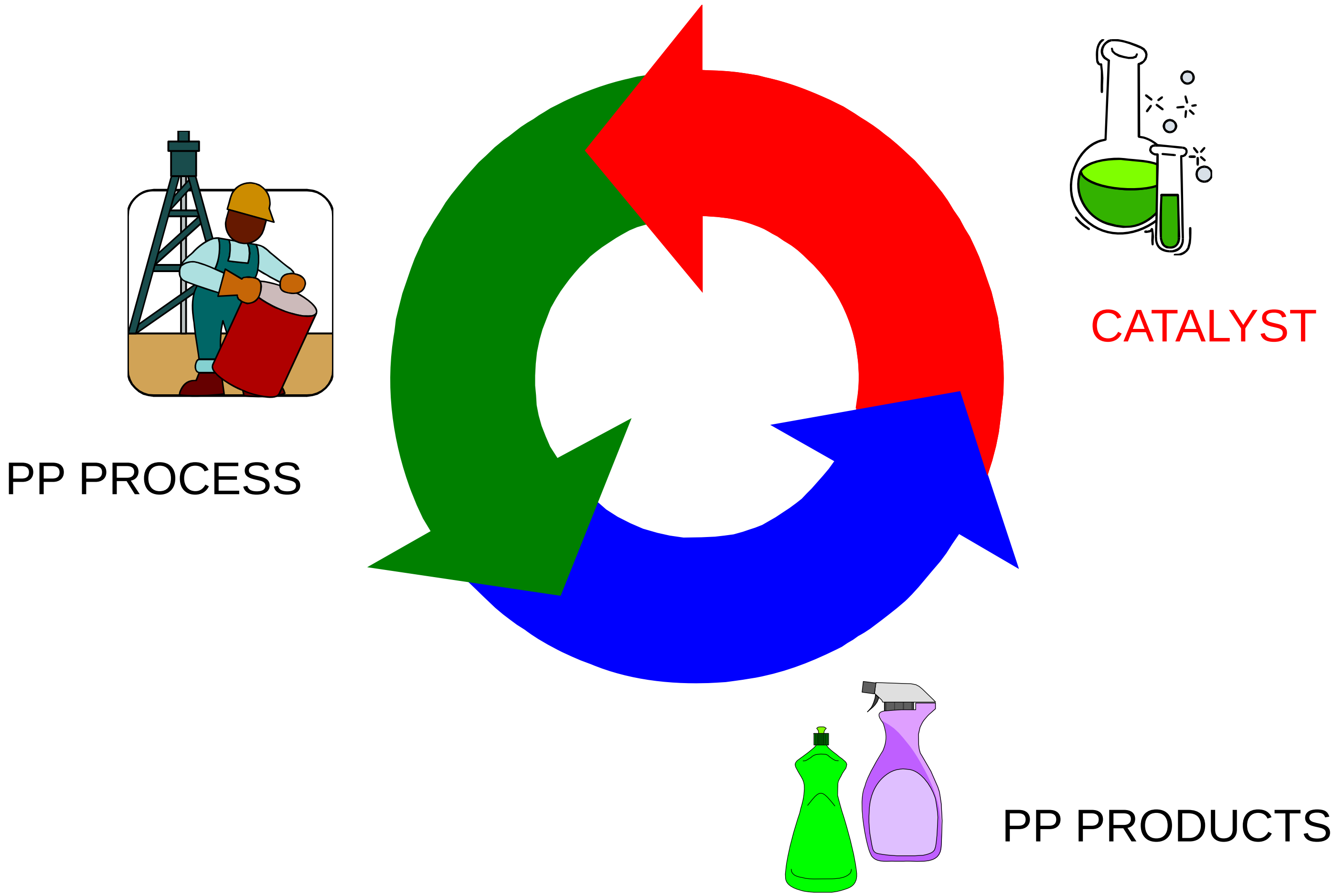
Polypropylene Industry



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Evolution of Polypropylene Catalyst

Gen	Catalyst	Activity	Isotacticity
1st	δ-$TiCl_3$.0.33$AlCl_3$ + DEAC	1500	90-94
2nd	γ-$TiCl_3$ + DEAC	4000	94-97
3rd	$TiCl_4$/Monoester/$MgCl_2$/ + TEAl/Ester	<20,000	90-96
4th	$TiCl_4$/Diester/$MgCl_2$ + TEAl/Silanes	>25,000	95-99
5th	$TiCl_4$/Diether/$MgCl_2$ + TEAl (Silanes ?)	>50,000	95-99
5 ½ (?)	$TiCl_4$/Other Non-phthalate Donors/$MgCl_2$ + TEAl + (Silanes ?)	>50,000	95-99
6th	Metallocene		

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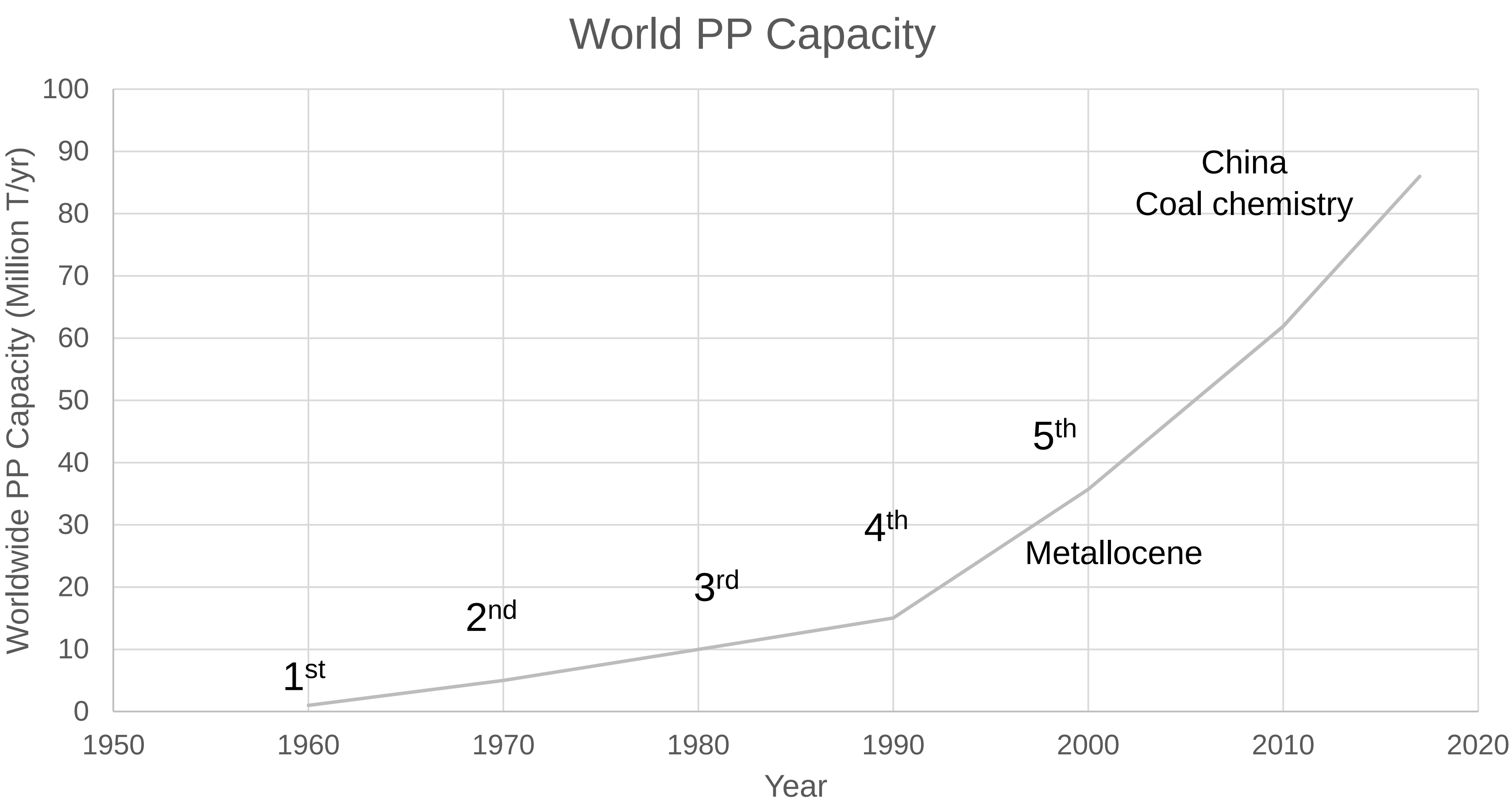
Evolution of Catalyst and Polypropylene Growth



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1st Generation Ziegler-Natta Catalyst

$TiCl_4$ + Al ⟶ $3TiCl_3$ + 1/3 $AlCl_3$

Ball Milling

+ Electron Donor ⟶ 1st Generation Z-N Catalyst

Co-catalyst: DEAC (di-ethyl aluminum chloride)

Most slurry PP plants were originally designed around this catalyst
Catalyst activity ~ 1500, isotacticity ~ 96%
Deashing and atactic removal required

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2nd Generation Ziegler-Natta Catalyst

$TiCl_4$ + Electron Donor —(Heptane)→ A (γ-$TiCl_3$)

A + DEAC → 2nd Generation Z-N Catalyst

Co-catalyst: DEAC (di-ethyl aluminum chloride)

Kazuo et.al, US 4,060,593

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2nd Generation Ziegler-Natta Catalyst

$TiCl_4$ + Electron Donor + DEAC —(Heptane)→ —(Filter/Wash)→ $TiCl_3ED$

+ DEAC + $C_3^{=}$ —(Heptane, Filter/Wash)→ Prepolymerized Catalyst

Co-catalyst: DEAC (di-ethyl aluminum chloride)

Abe et.al, US 4,200,717
Catalyst activity ~ 4000, isotacticity ~ 96%
Deashing and atactic removal required

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3rd Generation Ziegler-Natta Catalyst

$TiCl_4$ + Ethyl Benzoate —(Heptane)→ —(Filter/Wash)→ $TiCl_3EB$

$MgCl_2$ + $TiCl_3EB$ —(Ball Mill)→ Ti/Mg Complex

Ti/Mg Complex + $TiCl_4$ —(Heptane, Filter/Wash)→ 3rd Generation Catalyst

Co-catalyst: Triethyl Aluminum (TEAl)
p-ethyl anisate

Giannini et.al, GBR 1,387,890
Catalyst activity ~15000, isotacticity ~ 96%
Atactic removal required

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3rd Generation Ziegler-Natta Catalyst

$Mg(OEt)_2$
$TiCl_4$
Ethyl benzoate

$\xrightarrow[\text{Filter/Wash}]{\text{Solvent}}$ Ti/Mg Complex

Ti/Mg Complex + $TiCl_4$ $\xrightarrow[\text{Filter/Wash}]{\text{Solvent}}$ 3^{rd} Generation Catalyst

Co-catalyst: Triethyl Aluminum (TEAl)
p-methoxy ethylbenzoate

Goodall et.al, US 4,414,132
Catalyst activity ~20000, isotacticity ~ 96%
Similar catalyst still widely used in Unipol process

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4th Generation Ziegler-Natta Catalyst

$Mg(OEt)_2$
Alcohol
Carbon dioxide
→ (Solvent)
Mg Carbonate Solution

Mg Carbonate + $TiCl_4$ → (THF; Filter/Wash) Catalyst Support

Catalyst Support + $TiCl_4$ + Phthalate → (Solvent; Filter/Wash) 4th Generation Catalyst

Co-catalyst: Triethyl Aluminum (TEAl), Silanes

Arzoumanidis et. al., US 4,866,022

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4^{th} Generation Ziegler-Natta Catalyst

$MgCl_2$ + Ethanol → Catalyst Support

Particle forming

Solvent

Catalyst Support + $TiCl_4$ + Phthalate → 4^{th} Generation Catalyst

Filter/Wash

Co-catalyst: Triethyl Aluminum (TEAl), Silanes

Most widely used catalyst today

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5^{th} / $5+^{th}$ Generation Ziegler-Natta Catalyst

$MgCl_2$ + Ethanol → Catalyst Support
Particle forming

Catalyst Support
+ $TiCl_4$
+ Non-phthalate donors
→ Solvent, Filter/Wash →
5^{th} Generation Catalyst

Co-catalyst: Triethyl Aluminum (TEAl),
Silanes (optional)

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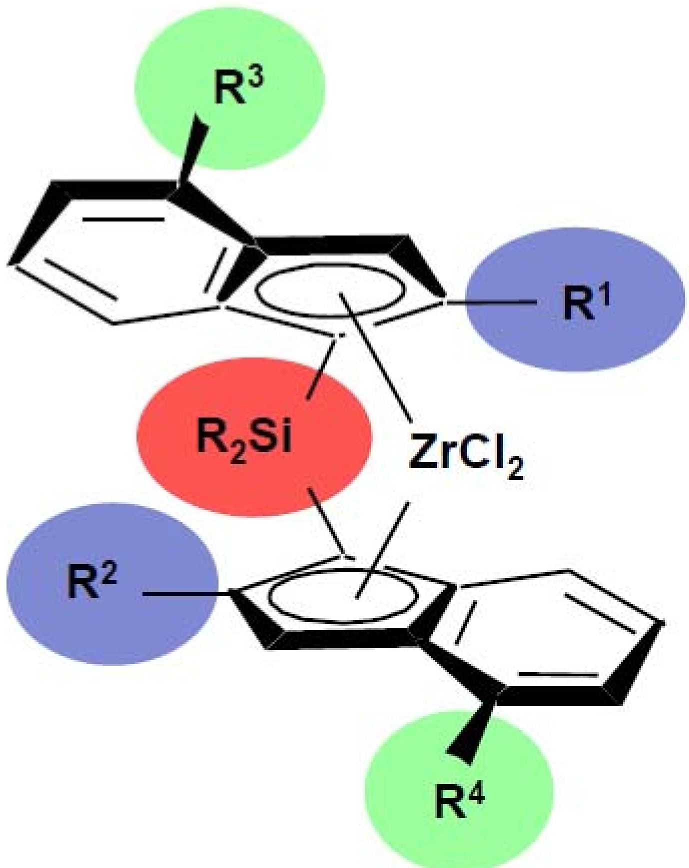
Metallocene and Single Site Catalysts



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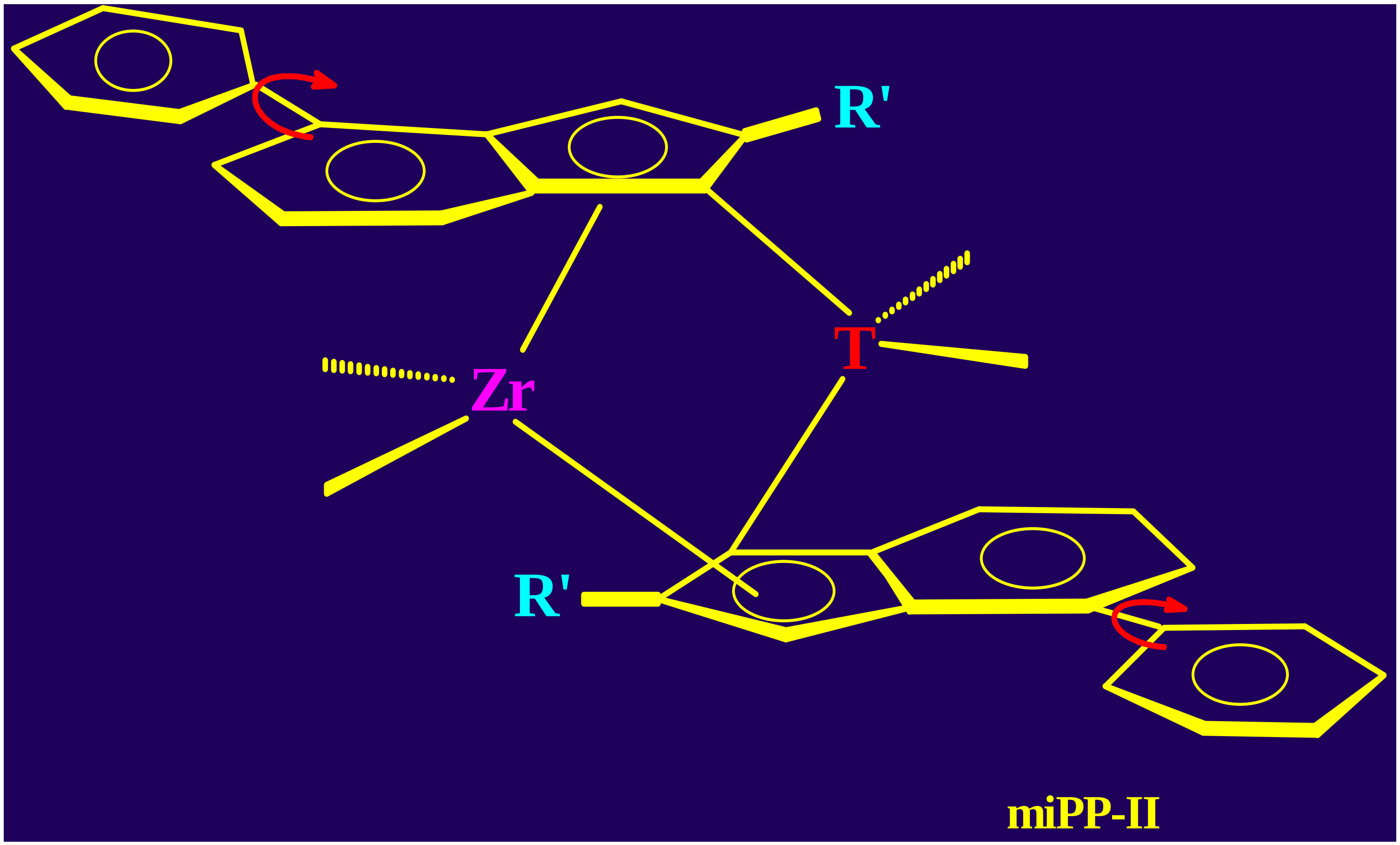
Metallocene and Single Site Catalysts



Metallocene for isotactic PP

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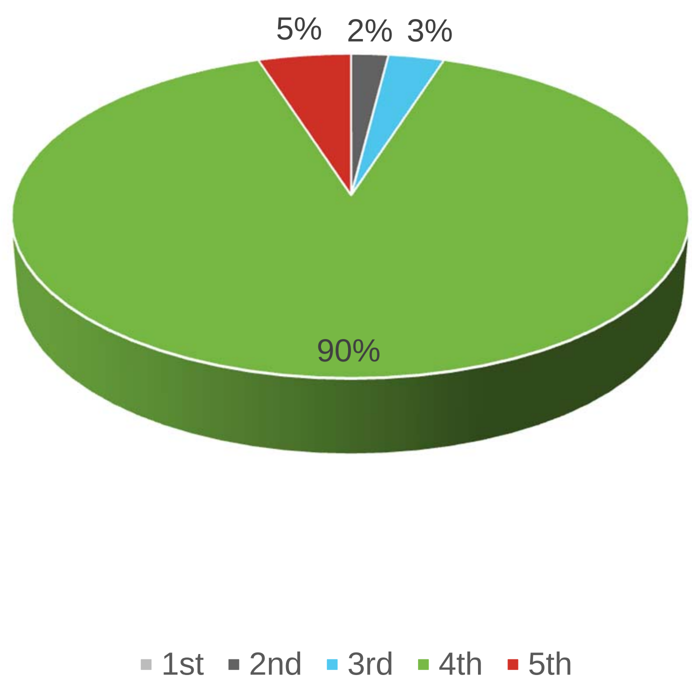
Catalyst Usage Status

Catalyst	Status	Producer
1st	No longer available on commercial basis	
2nd	Limited application in slurry plants	Grace (formerly BASF)
3rd	Still widely used in Unipol process	Grace Reliance (captive use)
4th	Most widely used catalyst	LBI, Grace, Clariant Sinopec Xiang Yang
5th	Applications in specialty grades	LBI, Clariant
5th +	Limited applications	Grace
Metallocene	Limited applications in specialty grades	ExxonMobil (captive use) JPP (captive use)

Demand by Catalyst Generation



Major Polypropylene Polymerization Processes

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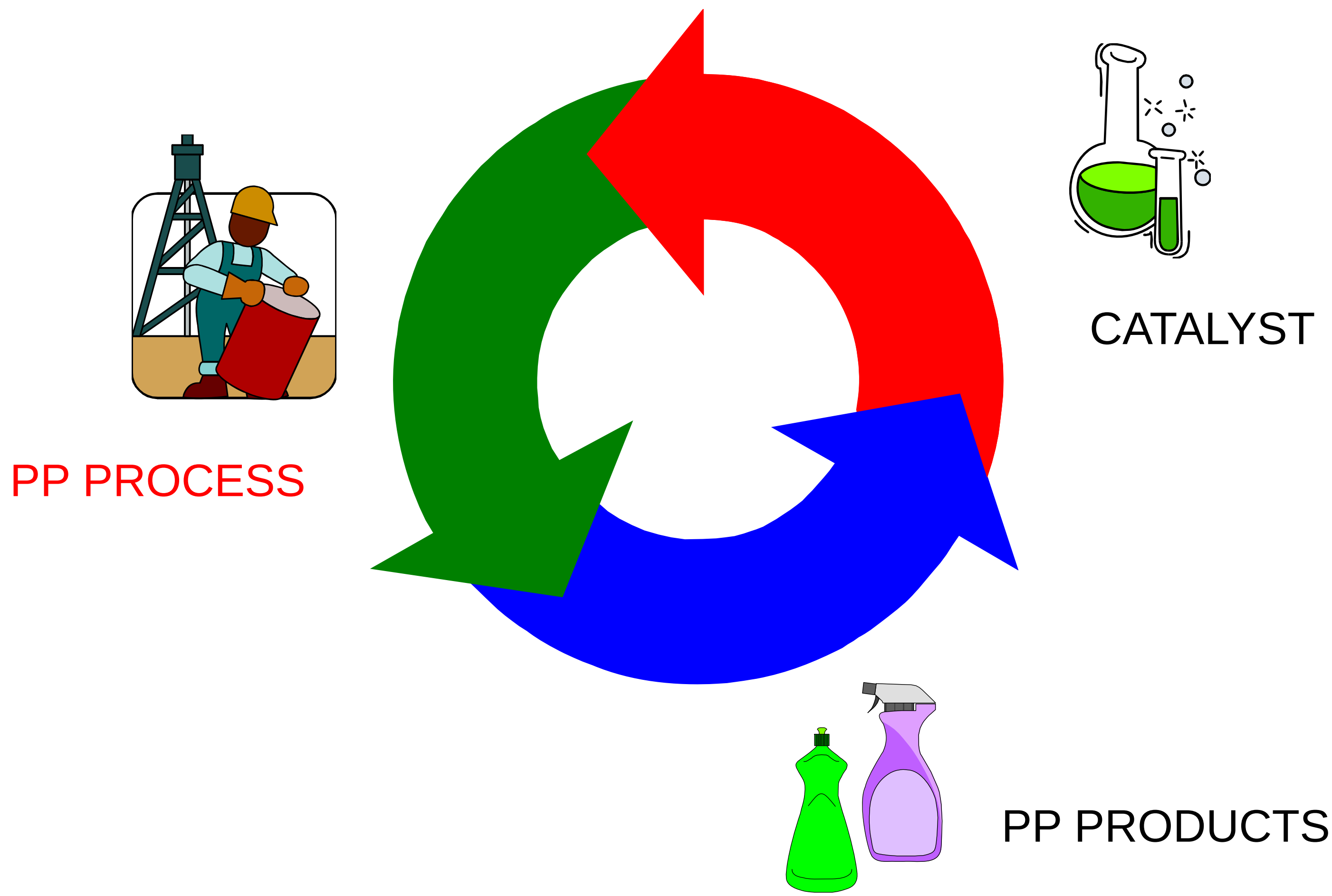

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Polyolefins: Reactors in Industrial Processes

– Provide an overview of different processes for which licensing information is available – some other processes out there with similar issues.

– What kinds of processes can we find?
  - Differentiated by
    - The phase in which the reaction occurs
    - The product we are making (e.g. PE vs PP vs Copolymers…)
    - Different Reactor Types
      - Gas phase.
      - Slurry/supercritical.
      - Different reactors for different processes

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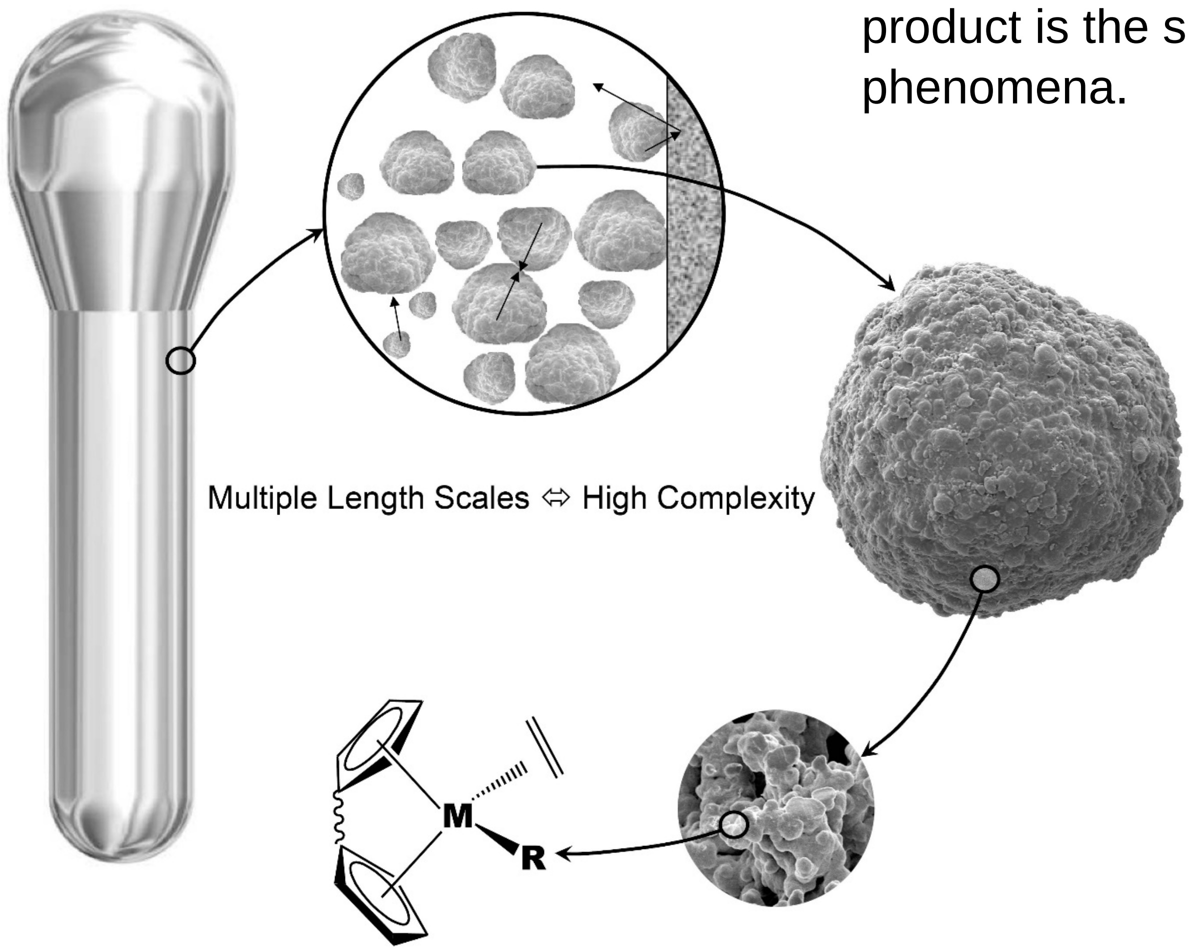
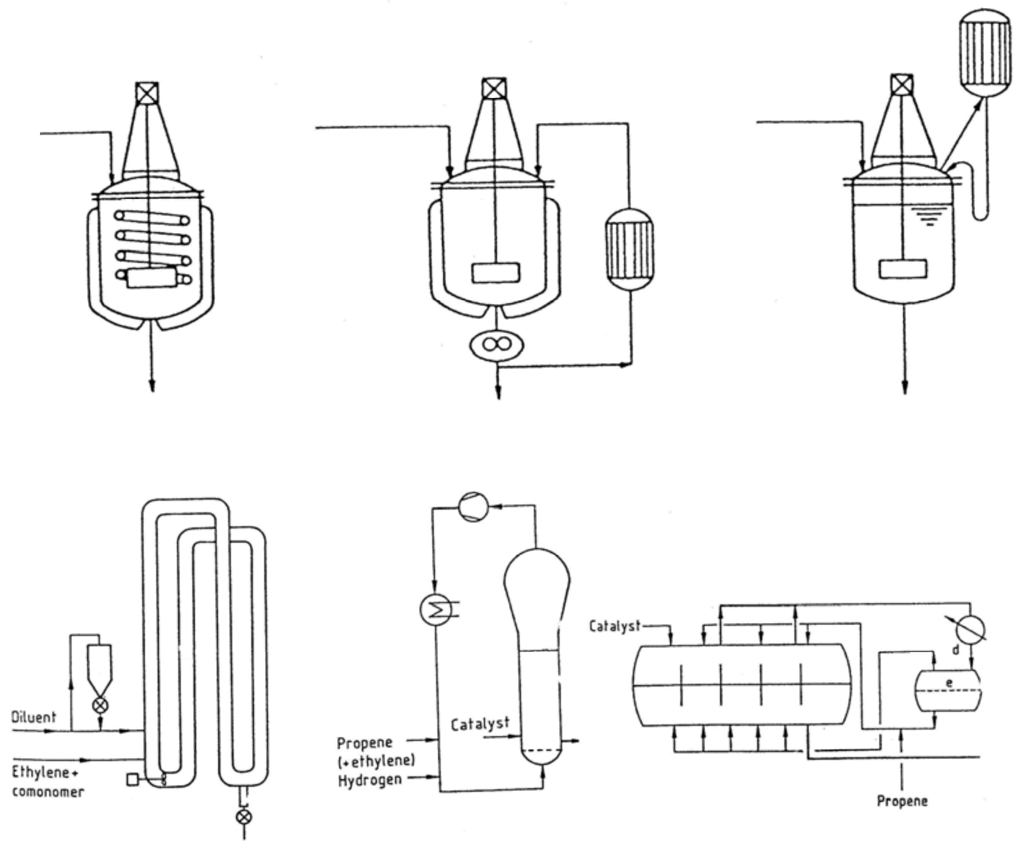
Polyolefins: Reactors in Industrial Processes

CONTEXT – Do not forget the multiplicity of length and time scales. The product is the sum of ALL of the phenomena.



Reactors and Processes

- Need to remove substantial quantities of energy from the process has led to different reactor configurations.
- Modern technology is moving away from standard agitated vessels (to some extent).
- Loop reactors, boiling beds, sprayed agitated vessels, fluidized beds… all allow production of up to 60-80 MT/h IN A SINGLE REACTOR.



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Reactors and Processes

– **HEAT REMOVAL (of course independent of chemistry):**

- **-**ΔHp= 800 kCal/kg for C_2H_4
- **-**ΔHp= 160 kCal/kg for PS
- Use extremely high throughput and low conversion per pass.

$$800\ \frac{\text{kCal}}{\text{kg}} \times 50{,}000\ \frac{\text{kg}}{\text{h}} \times \frac{1\,\text{h}}{3600\ \text{s}} \times \frac{1000\ \text{cal}}{\text{kCal}} \times \frac{4.184\ \text{J}}{\text{cal}} \approx \mathbf{50\,MW}$$

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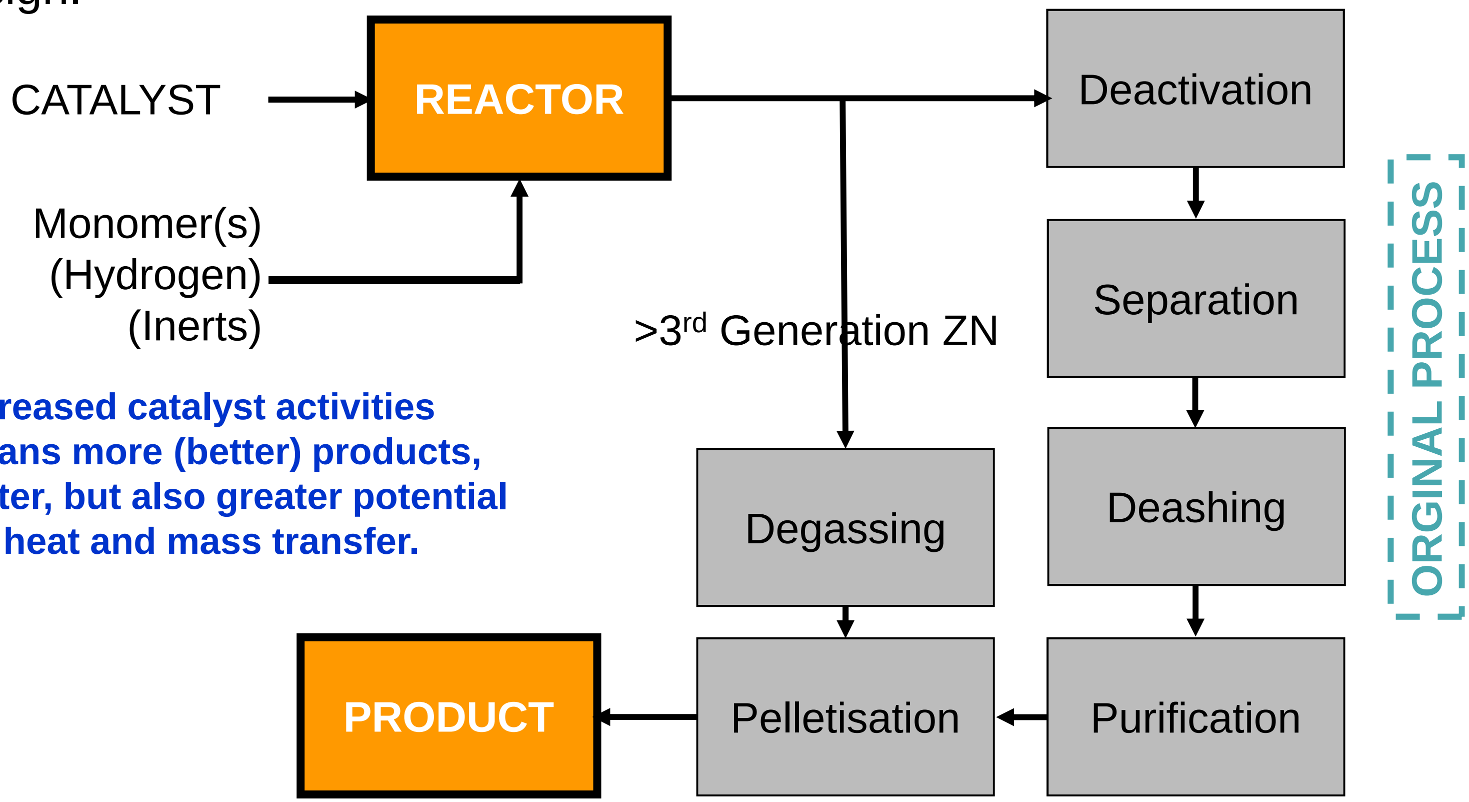
Reactors and Processes

– Developments in catalyst technology have driven process design AND reactor design.



Increased catalyst activities means more (better) products, faster, but also greater potential for heat and mass transfer.

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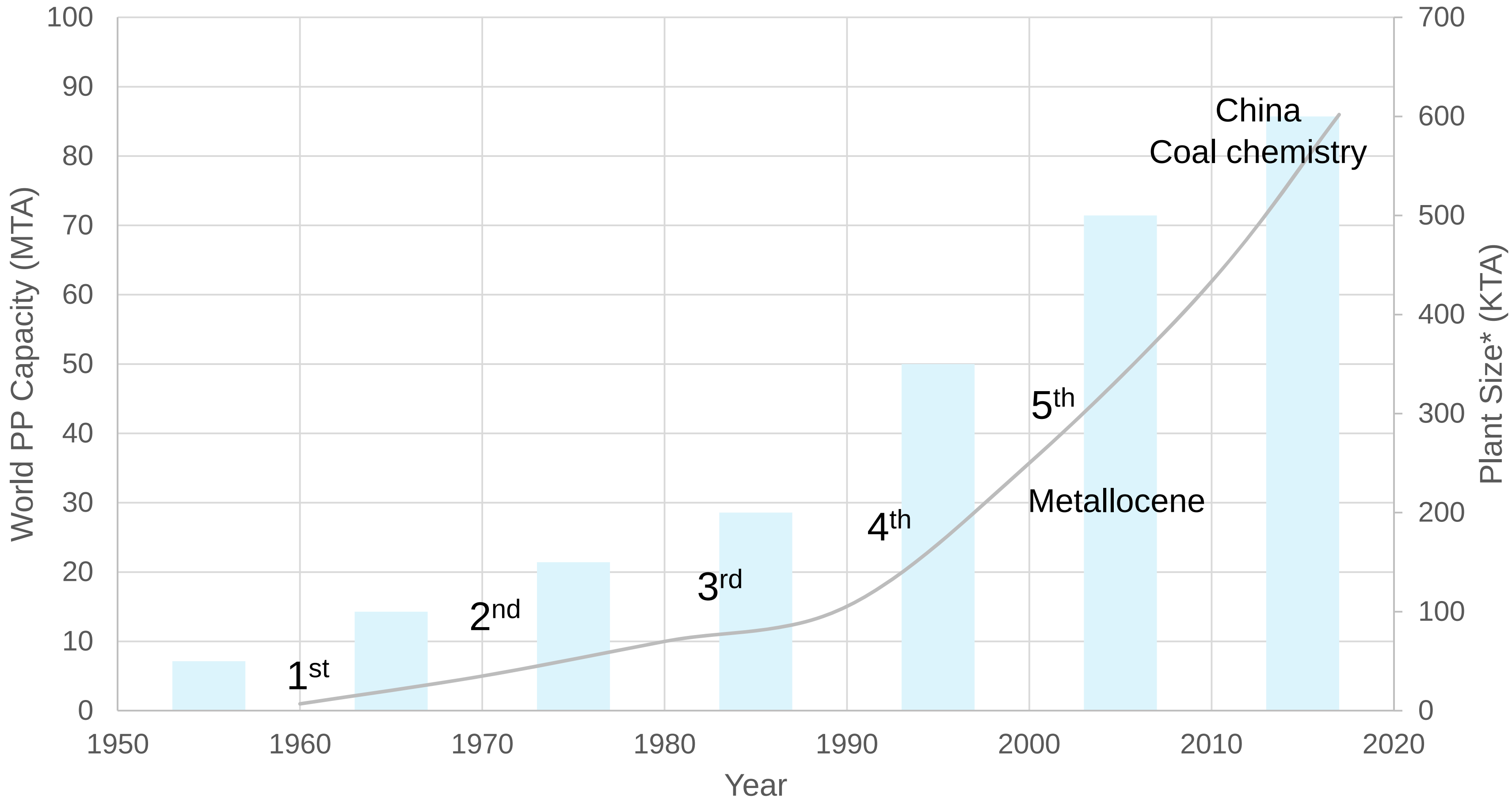
Reactor and Process
Size of World Scale PP Plant Continues to Grow



* Size of world scale plant

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Commercial PP Process Technologies

Slurry	Bulk	Loop/Gas	Gas
Montedison Hercules ICI Hoechst Mitsui Mitsubishi Mitsui-Toatsu Chisso	Hypol I (Mitsui) LIPP CP Chem Appryl (Total) Sumitomo Bulk	Spheripol (LBI) Hypol II (Mitsui) Sinopec II Borstar (Borealis)	Novolen (CB&I) Unipol (Grace) Innovene (Ineos) Horizone (JPP) Sumitomo Spherizone (LBI)

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Commercial PP Process Technologies

Process	**Reactor Type**	**Mode of Operation**	**Reactor Temperature**	**Reactor Pressure**	**Residence Time**
Hydrocarbon Slurry	Series of stirred autoclaves	Slurry	65 – 75 °C	8 – 12 bar	Up to 5 hrs
Hypol I (Mitsui)	Autoclaves	Liquid pool (bulk)	65 - 75 °C	30 - 35 bar	~ 2 hrs
Spheripol, Hypol II	Loop	Liquid pool (bulk)	65 - 75 °C	30 - 35 bar	~ 2 hrs
Novolen	Vertical stirred bed gas phase	Non-condensed gas phase	75 - 80 °C	30 - 35 bar	~ 1 hr
Innovene (Ineos) Horizone (JPP)	Horizontal stirred bed gas phase	Non-condensed gas phase	60 - 70 °C	25 - 30 bar	~ 1 hr
Unipol	Fluidized bed gas phase	Condensed gas phase	60 - 70 °C	25 - 30 bar	~ 1 hr
Sumitomo	Fluidized bed gas phase	Non-condensed gas phase	60 - 70 °C	25 - 30 bar	~ 1 hr

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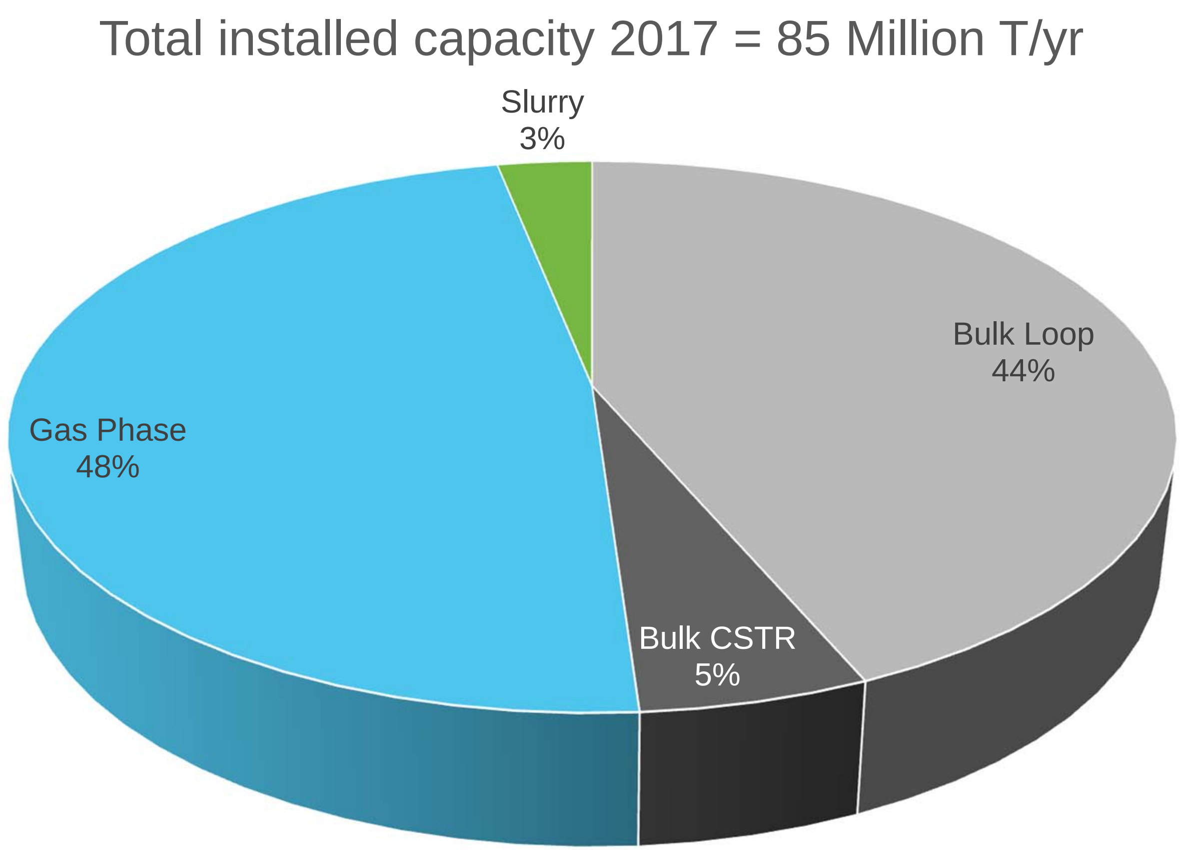
PP Worldwide Installed Capacity
Process Choice



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Hydrocarbon Slurry Processes

- 1st generation polypropylene process utilizing 1st generation Ziegler Natta catalyst
- Many slightly different processes (using various hydrocarbon diluents) developed by many companies: Montedison, Hercules, ICI, Mitsui, Mitsui-Toatsu, etc.
- Usually consists of a series of autoclaves to extend residence time due to low catalyst activity
- Usually require de-ashing of catalyst residue due to low catalyst activity
- Atactic polymer dissolves in the diluent and has to be removed
- Most slurry plants have been shut down due to poor economics
- Remaining plants focus on specialty polymers,

  e.g. low catalyst residue capacitor film
- Most slurry plants have switched to 4th generation catalysts

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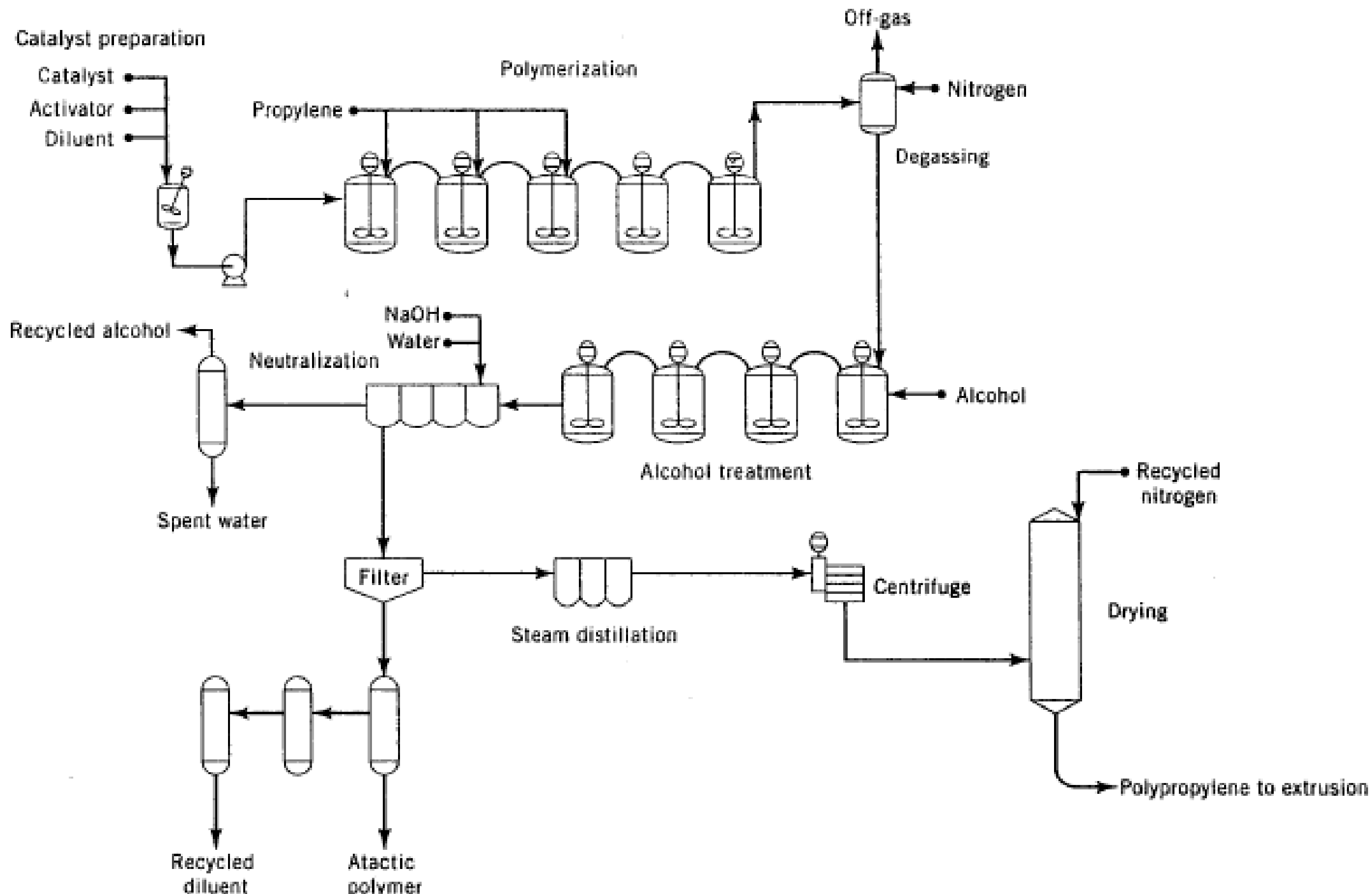
Slurry Process



Hercules HCPP process

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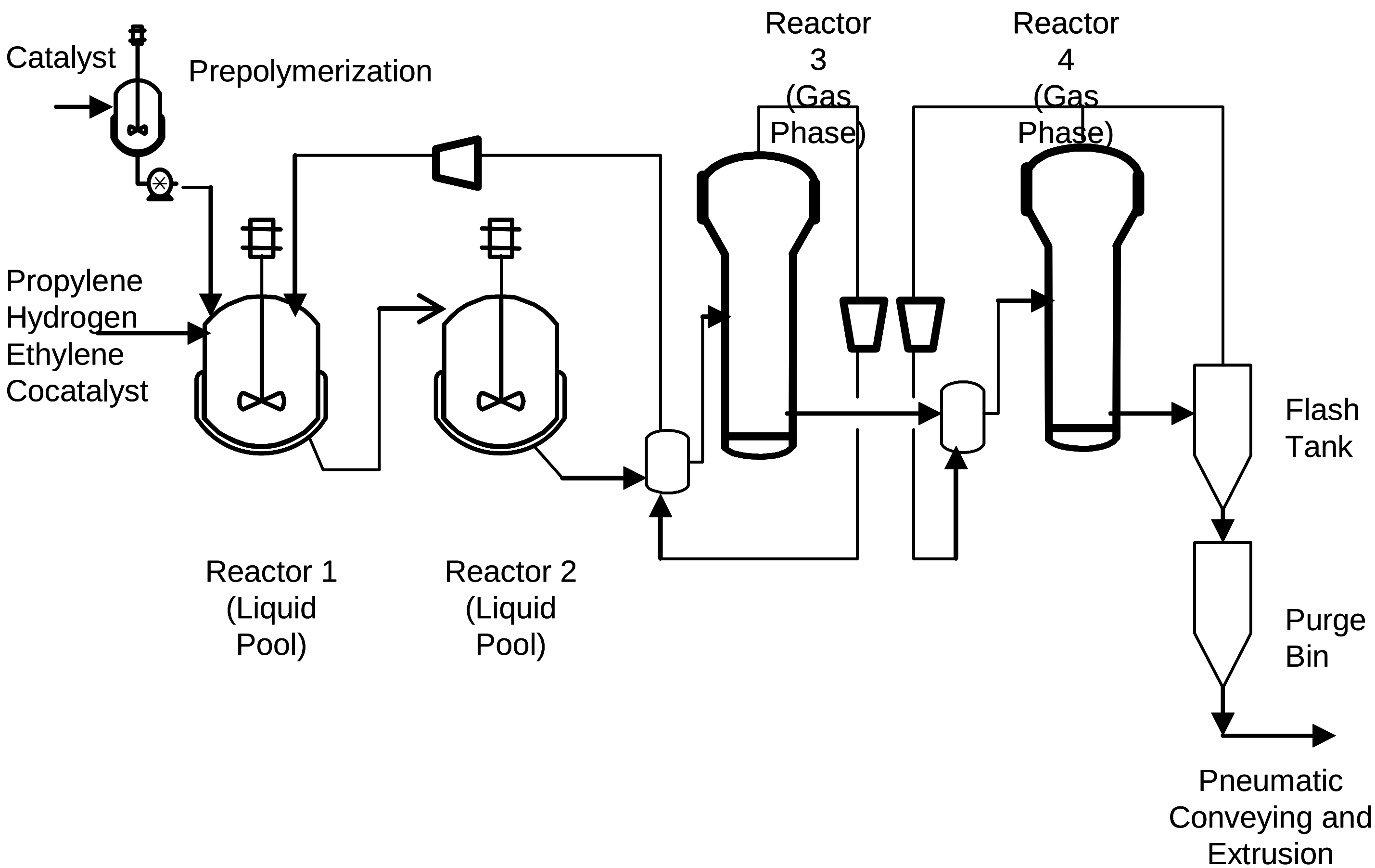
Bulk Process – Mitsui Hypol I

- Developed and licensed by Mitsui in the 1980's
- Consists of 2 stirred autoclaves in series, then 2 fluidized bed gas phase reactors for impact copolymers
- Some plants have only 1 autoclave and one gas phase reactor
- Autoclave is operated in liquid pool (bulk) condition
- Due to the multi-reactor configuration, can produce impact copolymer with broad MWD for better impact-stiffness balance
- Catalyst is prepolymerized in a batch system with low degree of prepolymerization

Mitsui Hypol I Process



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Bulk Process Variations

– LIPP Originally developed by Shell
  - Homopolymer only
  - 4 plants still operational
  - Single stirred bulk reactor

– Appryl (now TOTAL)
  - Homopolymer only
  - Single stirred bulk reactor
  - Several plants still in operation

– Local Chinese Bulk processes
  - Homopolymer only
  - Some operating in batch mode, typically without pelletization

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Spheripol Process

– Can easily make over 400 kT/yr – production rate limited only by pelletizing section of plant (and sales potential!).

– Can be configured with 1 or 2 loop reactors (2-6 legs) for PP homopolymer in bulk, or with additional train of FBRs (mostly one, some have two)

– Loops run at 70 – 75 °C, 40-45 bar; typical catalyst productivities on the order of 35 – 50 TPP/kg catalyst

– Residence time in two loops about 1.3-1.5h and solids are at about 55% (limited by flowability of slurry)

– Gives the possibility of making full range of MWD and copolymer compositions, with up to 45% rubber in the 4 reactor process

– FBRs (for ICP) typically at 10-15 bar, 75-80°C

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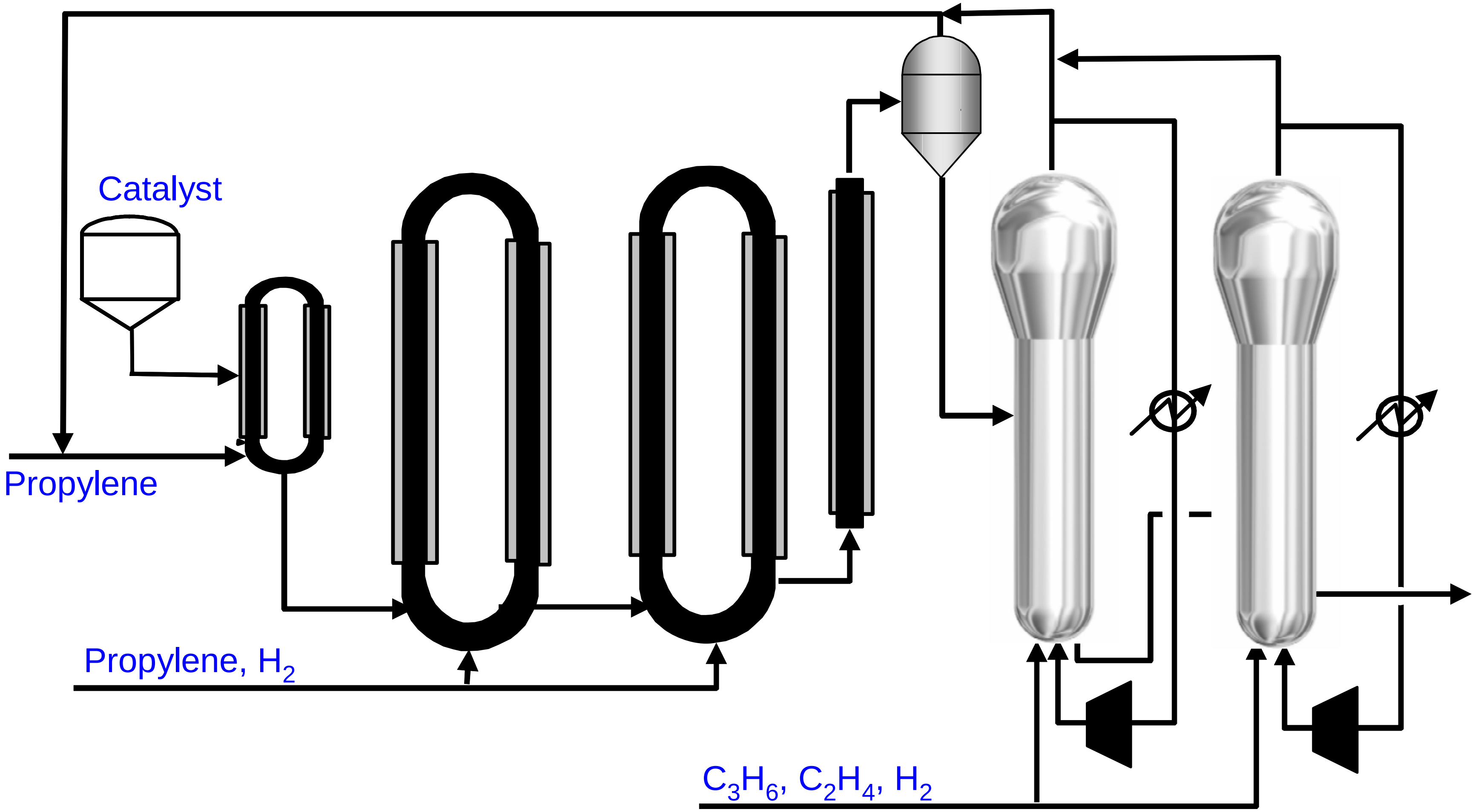
Spheripol Process



Bulk Loop Process Variation – Borealis Borstar

- Similar structure to BORSTAR PE where the loop reactor is (supercritical) propylene instead of propane
- Loop operated at 80-100°C, pressure adjusted accordingly from 50-60 bar.
- High P in loop means can have relatively high hydrogen concentrations and wide range of MFR (wider than others).
- Get good utilization of propylene since unreacted propylene from loop is apparently fed to gas phase reactor ➔ vaporization contributes to heat removal in GPR
- Depending on catalyst can get productivities of 100000 g/g on highly active ZN catalysts
- Number of gas phase reactors depends on type(s) of product to be made (e.g. 25% total rubber with one, 50% with two).
- GPR (1) run at 80-100°C, and P between 22-35 bar; GPR (2) at 75-90°C and 15-25 bar.

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Bulk Loop Process Variation – Borealis Borstar

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- Loop operated at 80-100°C, pressure adjusted accordingly from 50-60 bar.
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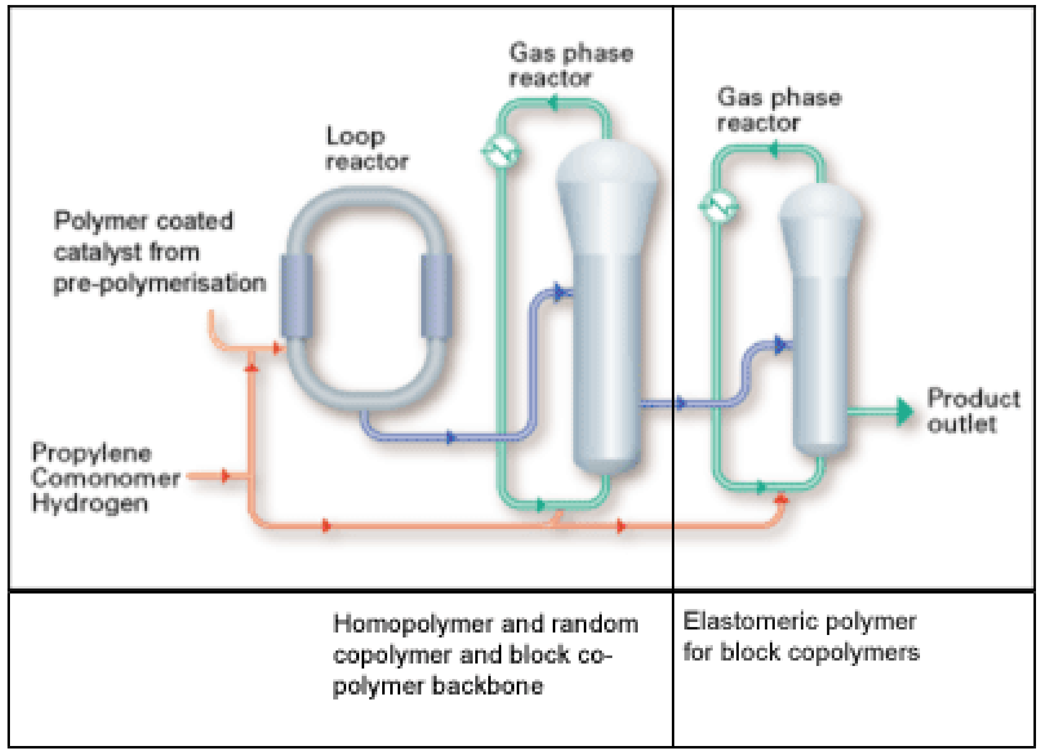
- Get good utilization of propylene since unreacted propylene from loop is apparently fed to gas phase reactor ➔ vaporization contributes to heat removal in GPR



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Bulk Loop Process Variations - Others

– Chevron Phillips	Based on CP Chem PE technology, one plant in operation
– Sinopec and local Chinese clones	Loop reactors based on Spheripol technology Many plants (~ 25) in operation in China
– ExxonMobil	Loop reactors based on Spheripol technology

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Gas Phase Processes

Process	Licensor	Reactor Configuration
Unipol	Grace (formerly Dow and Union Carbide)	Fluidized bed
Novolen	CB&I Novolen	Vertical stirred bed
Innovene	Ineos (formerly BP Amoco)	Horizontal stirred bed
Horizone	Japan Polypropylene (formerly Chisso, developed together with Amoco)	Horizontal stirred bed
Sumitomo	Captive use only, not licensed	Fluidized bed
Spherizone	Lyondell Basell (LBI)	Multi-zone reactor + Gas Phase for ICP

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PP in Gas Phase - Reactors and Processes
UNIPOL PP (Grace / Dow Chemicals)

- Essentially for PP and PP-C_2 random copolymers with up to 6 % ethylene and some butene.
- Impact copolymers with up to 37% EPR (22% ethylene)
- Catalysts are added in a mineral oil slurry.
- Average residence time of 1 hour
- Capacity of 260 kT/yr for impact copolymers and 400-500 kT/yr for homopolymers
- Some condensed cooling with propylene and propane.

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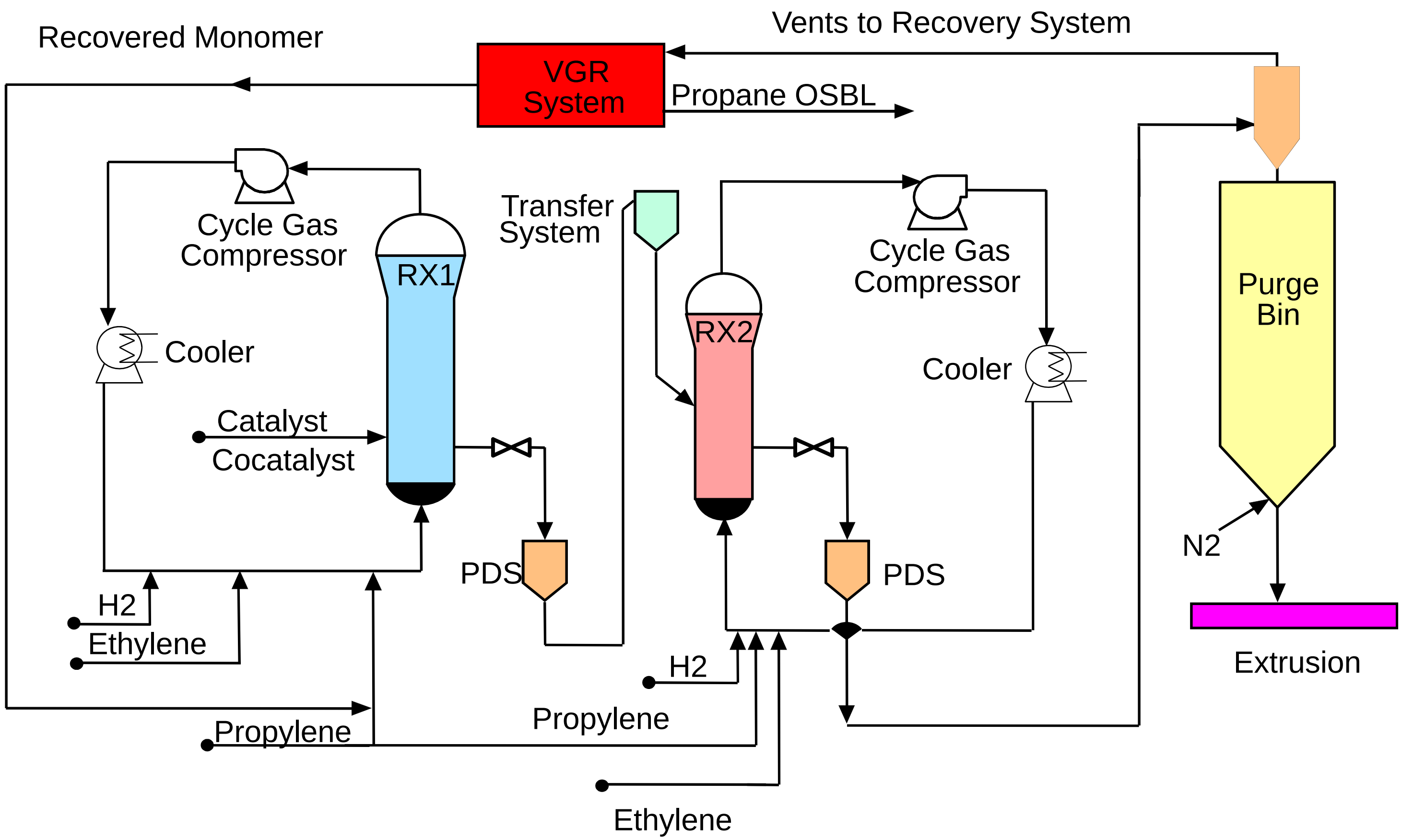
Unipol Process



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PP in Gas Phase - Reactors and Processes
NOVOLEN (CB & I)

- Very successful process due to its simplicity of operation (and types of catalyst available).
- Earlier processes small, flexible operations of 80 kT/yr, but can go up to 600 kT/yr with highly active ZN catalysts.
- 2 stirred powder beds in series, up to 125 m^3 each.
- T $\in$ 70-80°C, P $\in$ 22-32 bars.
- Heat is removed by evaporating liquid C_3 in reactor and condensation in external cooler. Recycle flow approx. 5 times of the production
- Can make homopolymer and random (1 or 2 reactors) or impact copolymers with 2 reactors.
- Transfer from R1 to R2 by pressure gradient.
- Impact copolymers can contain upwards of 50% EPR.
- Metallocene catalysts are available for this process.

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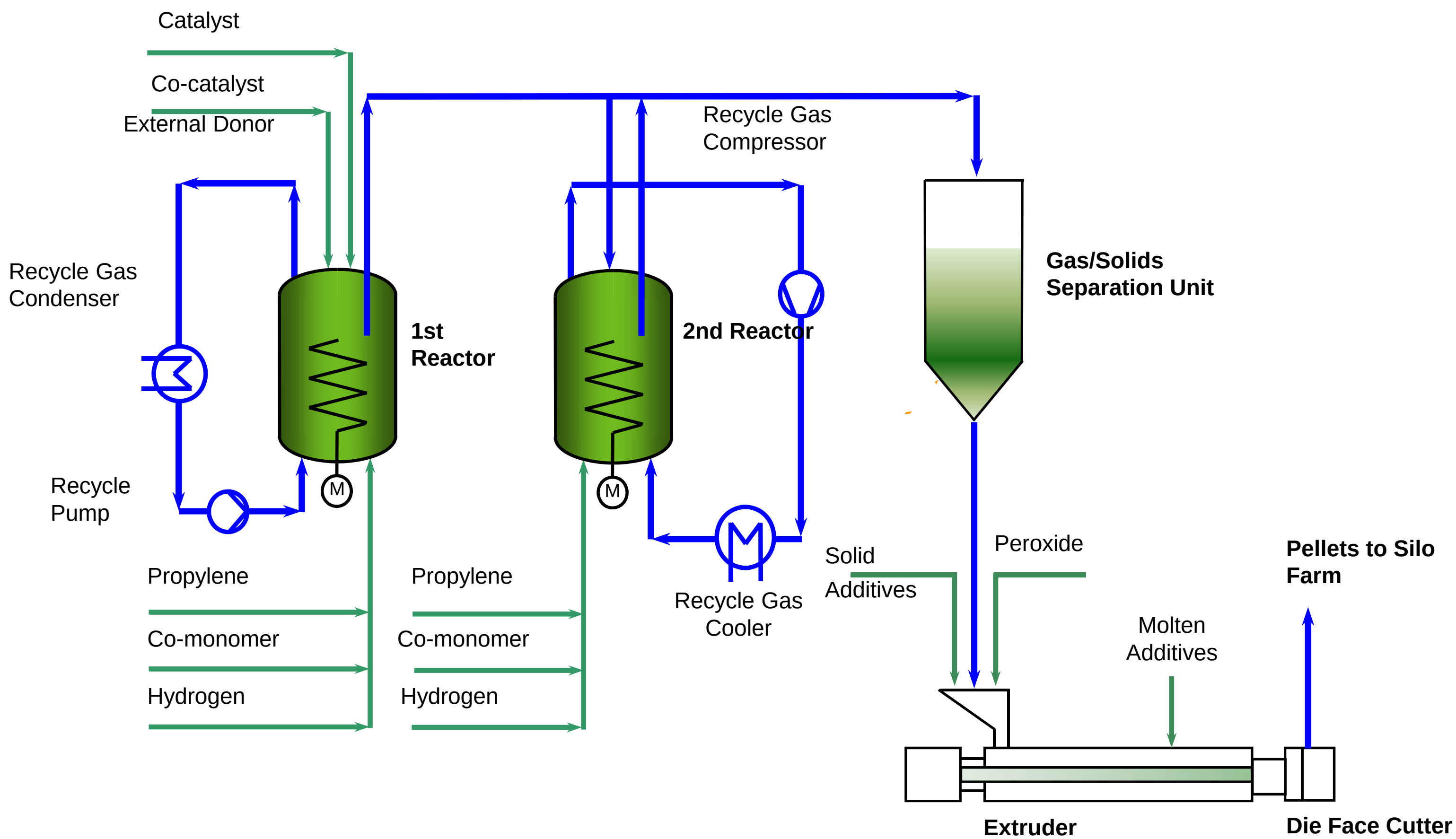
Novolen Versatile Reactor Concept (Novolen VRC™) System



The two reactors can be operated in parallel or cascade mode for the production of homo polymers, random co-polymers, impact co-polymers and TPOs

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PP in Gas Phase - Reactors and Processes

Innovene and Horizone

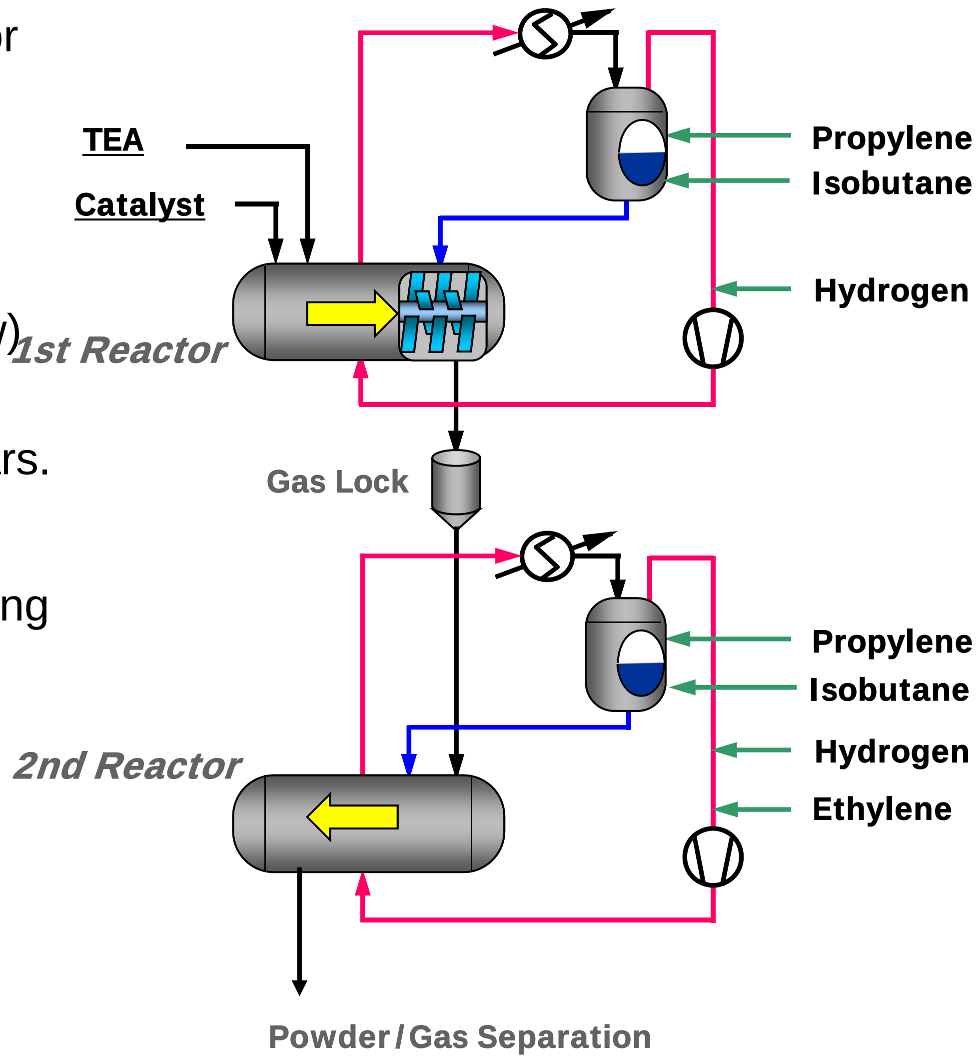
- Single agitated horizontal stirred bed, or cascade for random or impact copolymers.

- RTD of the beds means that grade transitions are very rapid (PFR-like flow).

- Conditions typically 65-85 °C, 20-25 bars.

- Can make polymers with more interesting and varied compositions in one reactor than with an FBR (or other type of continuous reactor).

- Horizone arrange their reactors one above the other; Innovene uses a powder transfer system.



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PP in Gas Phase - Reactors and Processes
Sumitomo

– Sumitomo also uses a 2-3 FBR cascade for homo- and copolymer grades.

– Copolymer reactors smaller than homopolymer ones (expected given relative Rp).

– Claim high crystallinity homopolymers due to the proprietary catalyst they design and sell directly to licensees.

– Have much smaller plant capacity (biggest is 270 kT/yr).

PP in Gas Phase - Reactors and Processes

SABIC Europe **(DSM) alternative reactor**

FBR with a draught tube

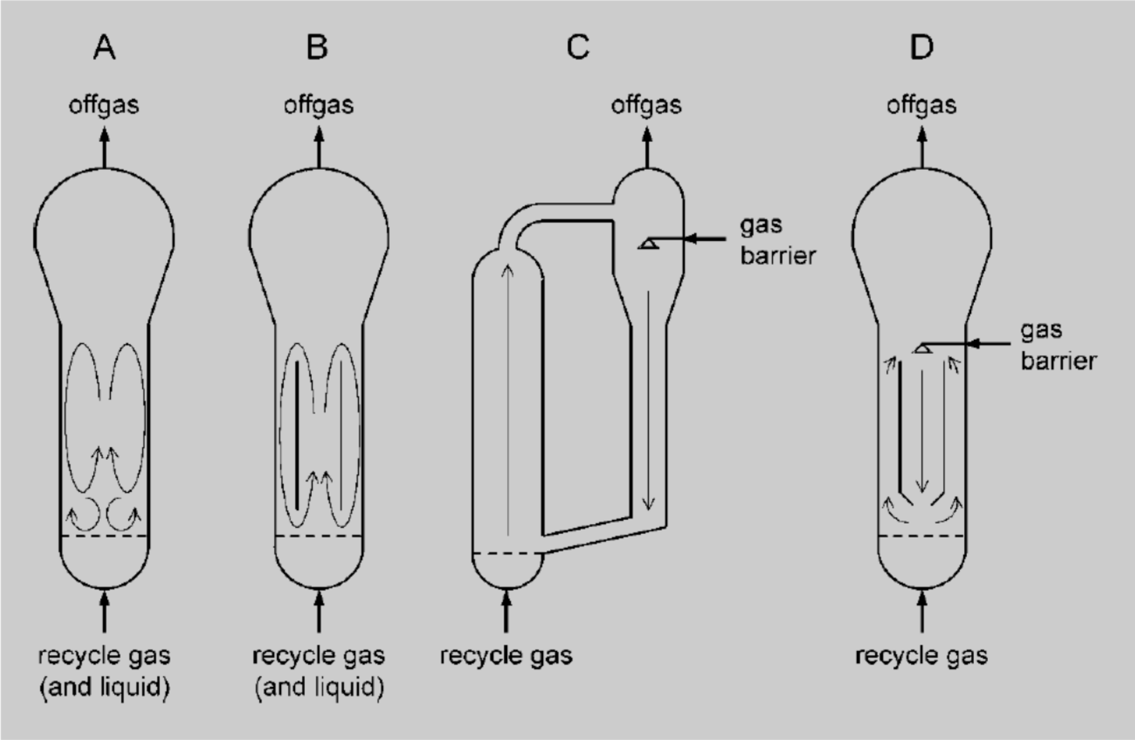

Figure. Types of fluidized bed reactors (FBR) for polyolefin production: A) Standard FBR, B) FBR with draft tube, C) Multi-zone circulating reactor and D) 2-zone draught reactor.

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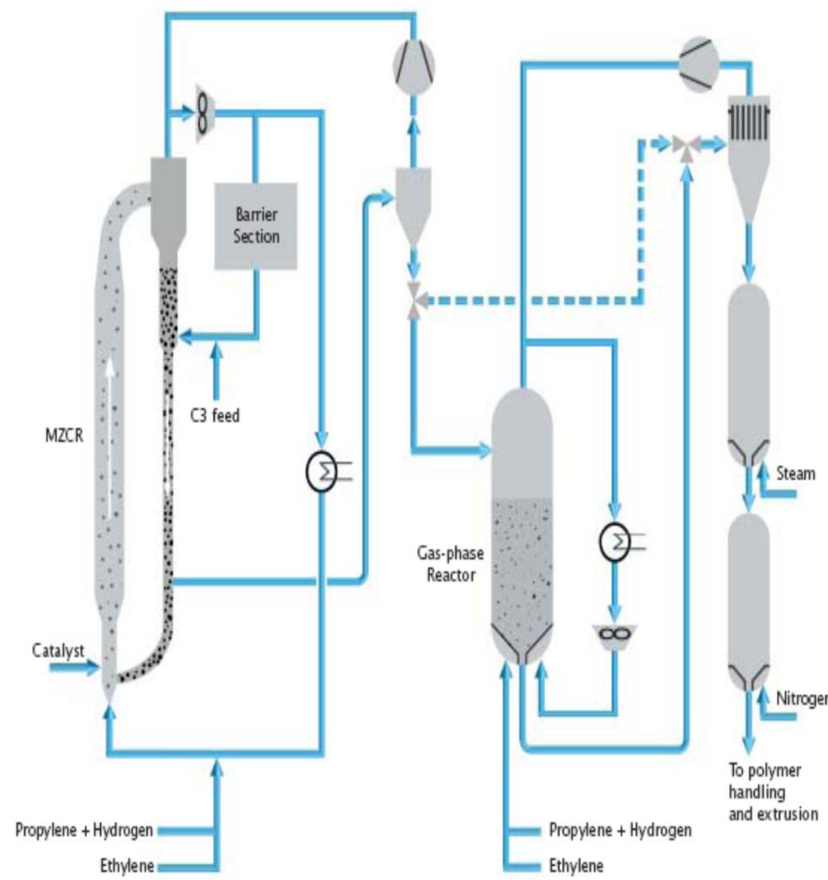
PP in Gas Phase - Reactors and Processes
Spherizone (LyondellBasell)

- Commercial operation began in 2000
- Can run with only MZCR, or with a second FBR for additional products (EPR for impact).
- MZCR is a riser downer with a velocity high enough that the polymer particles are entrained and the entire bed moves to the 2nd zone (downer).
- Downer works like a packed-bed reactor.
- Some publications mention prepolymerization zone… never shown on process flow diagrams.
- Different compositions in different zones offer possibility of making layered, intimately mixed products in ONE reactor.
  - Different MWD distributions
  - Different levels of crystallinity

Spherizone process



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Propylene Polymerization Reactors: Summary

Reactor Configuration	Advantages	Disadvantages
Stirred autoclave	• Low capital cost	• Low heat transfer area • Low space time yield
Loop	• High heat removal • High space time yield • Stable operation	• Limited flexibility
Fluidized gas phase	• Low capital cost • High heat removal • High space time yield	• More difficult to operate • Grade transition difficult due to large size.
Vertical stirred gas phase	• Low capital cost • Reactors can be used in parallel to increase throughput • Broad operating window • High fines tolerance	
Horizontal stirred gas phase	• PF characteristic allows for fast grade transfer	• More complex reactor design, higher capital cost.
Circulating Bed	• Better polymer properties control • Different reacting zone allows production of product not possible with traditional reactors	• Complex reactor design, high capital cost • More difficult to operate due to its complexity

Adapted from Soares, McKenna, Cheng, Polyolefin Reaction Engineering, in *Polymer Reaction Engineering,* J.M. Asua (ed.), Blackwell, 2007

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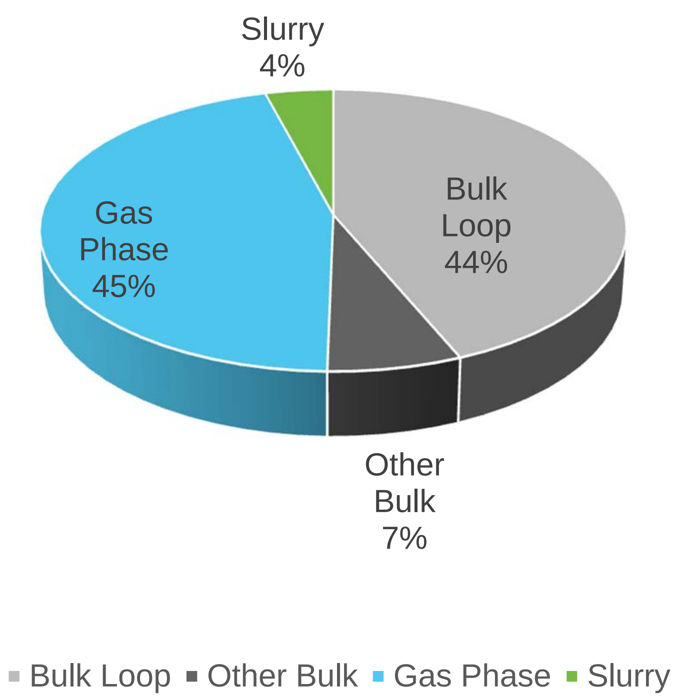
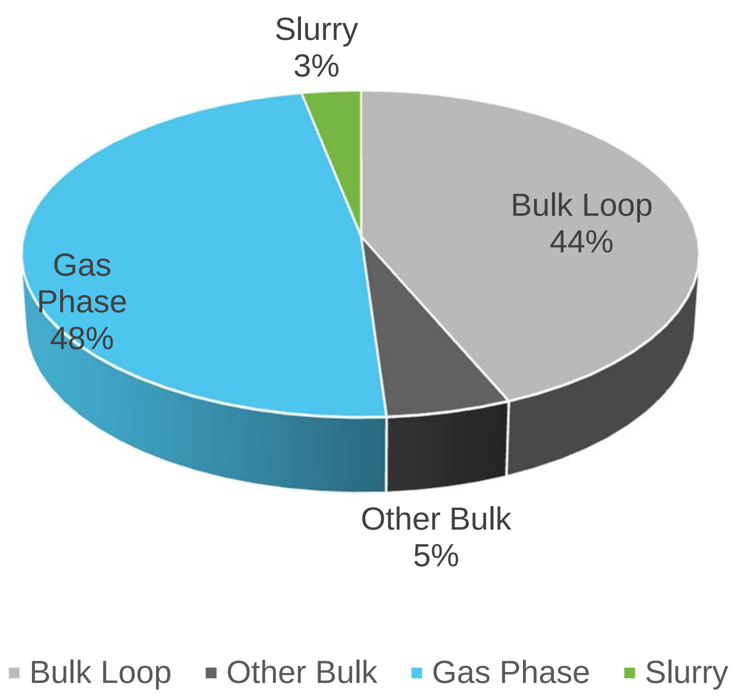
PP Process Choice

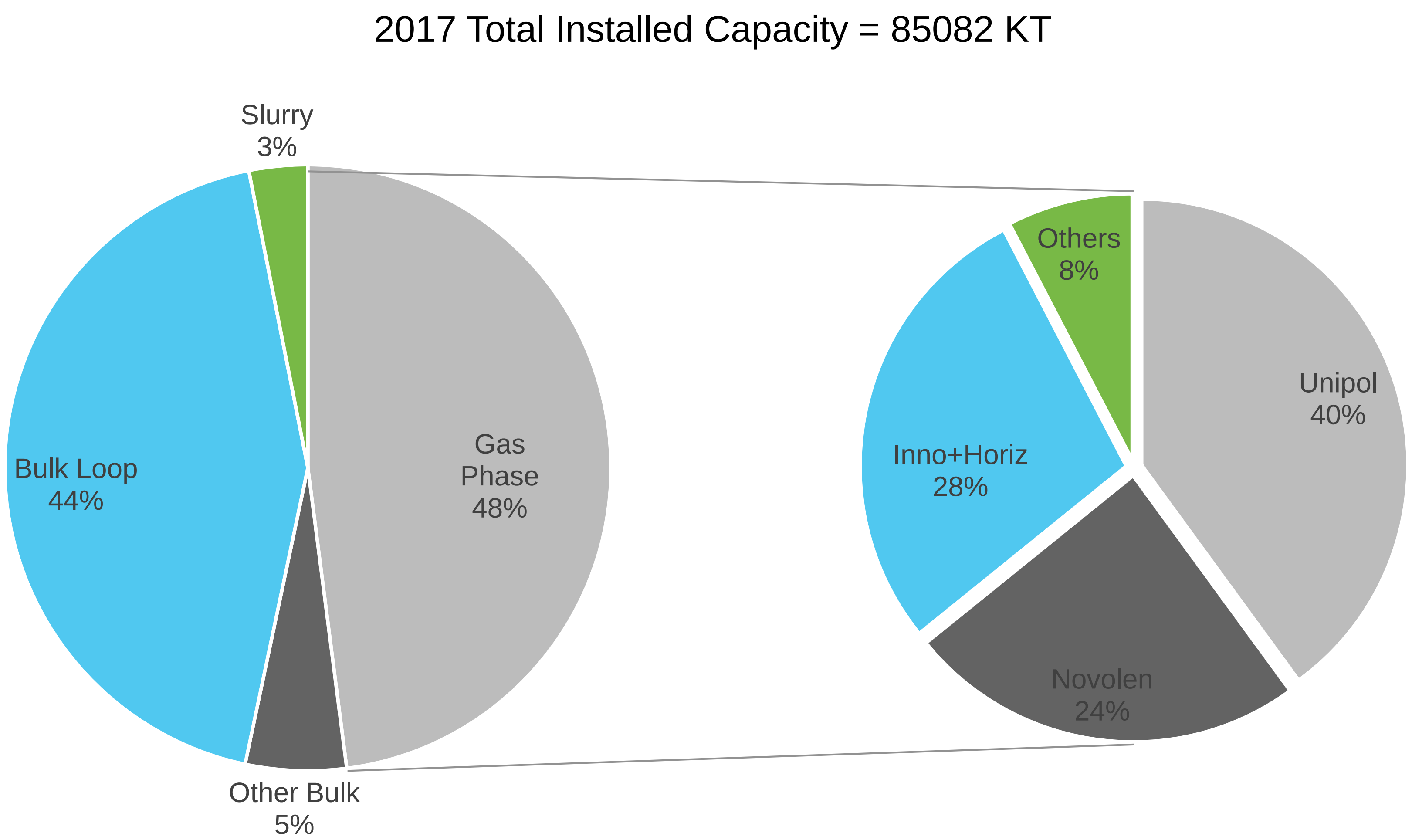
2012
Total Capacity = 63862 KTA



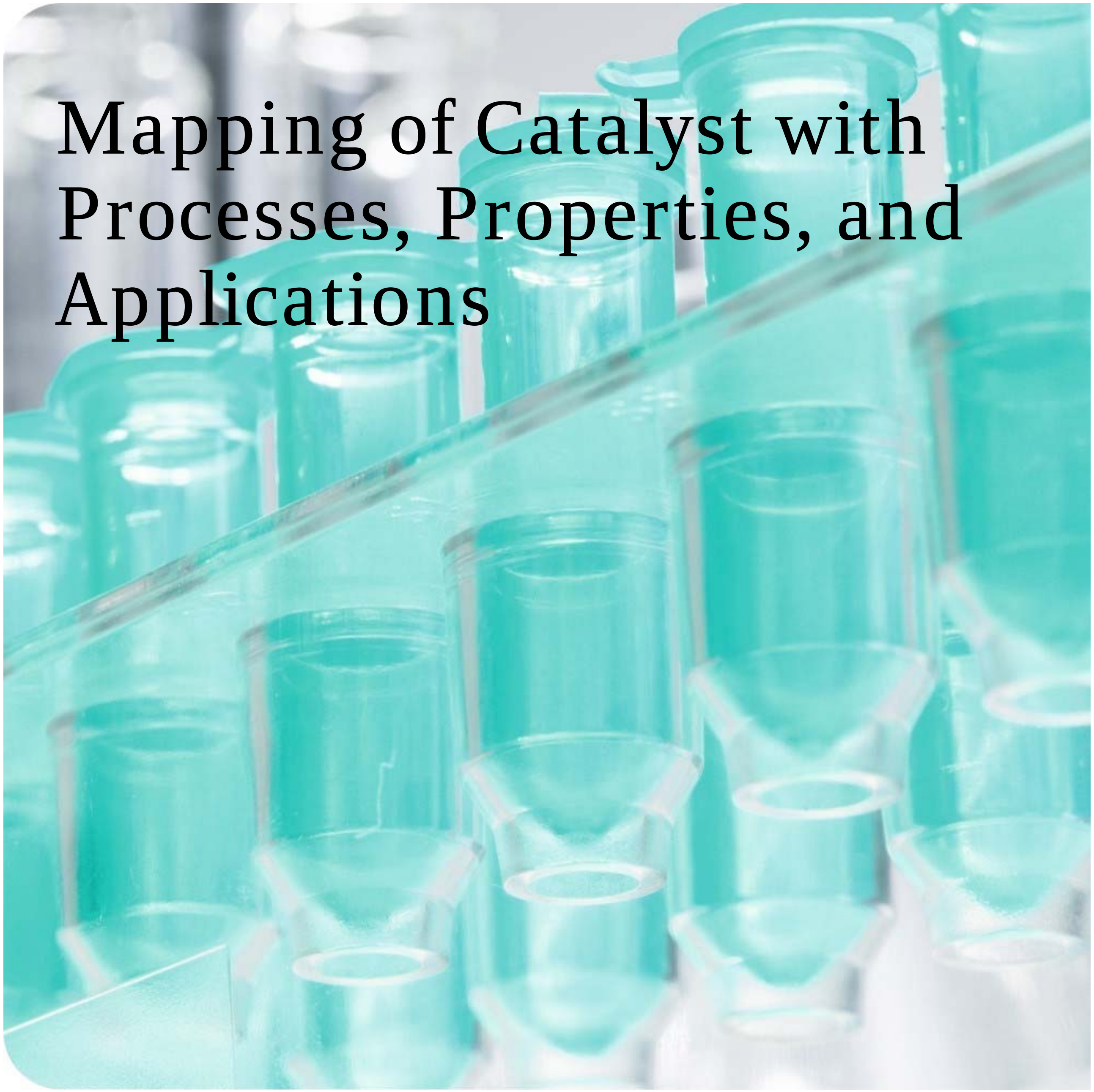
2017
Total Capacity = 85082 KTA



PP Process Choice



Mapping of Catalyst with Processes, Properties, and Applications



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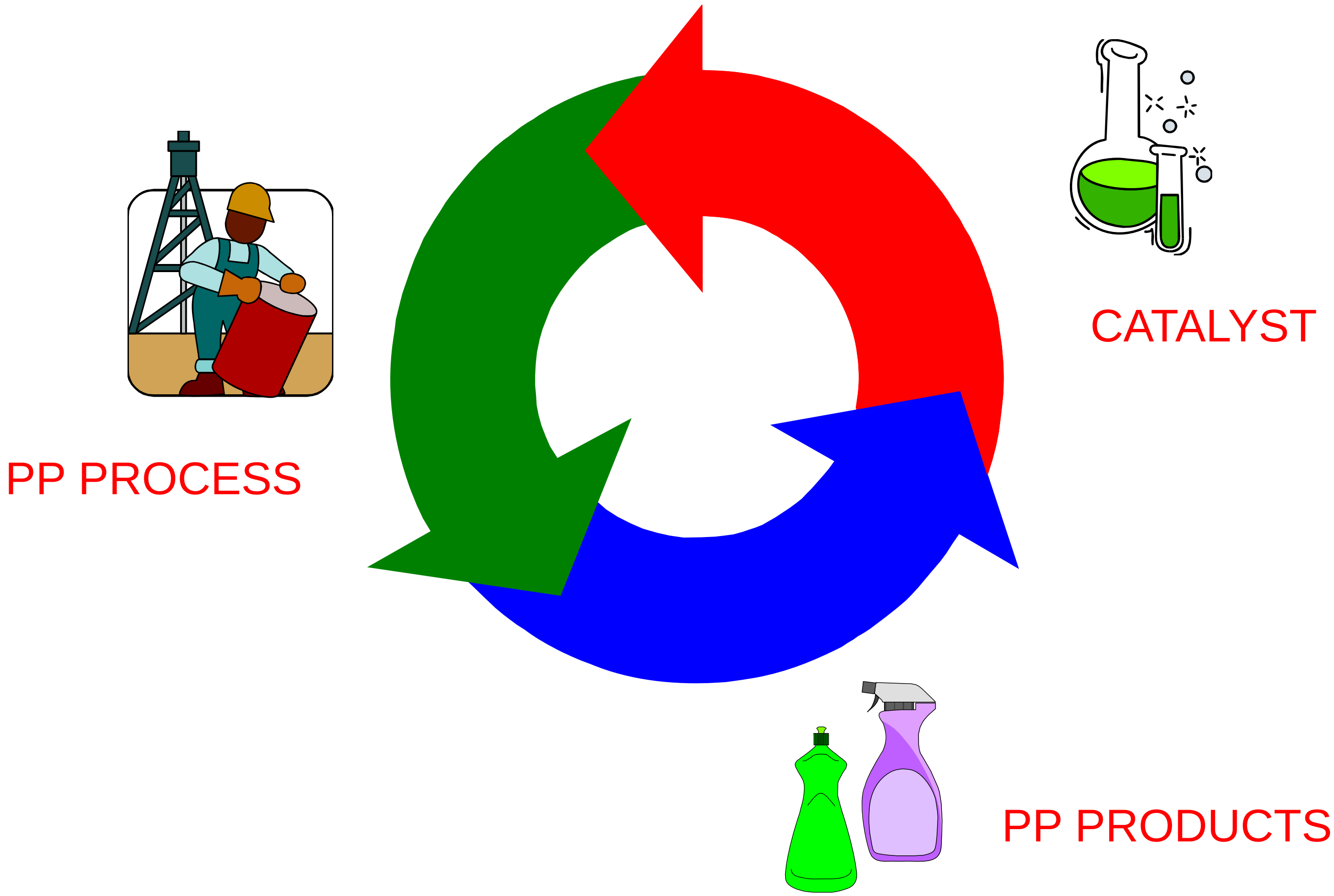
what is precious to you?

Polypropylene Industry



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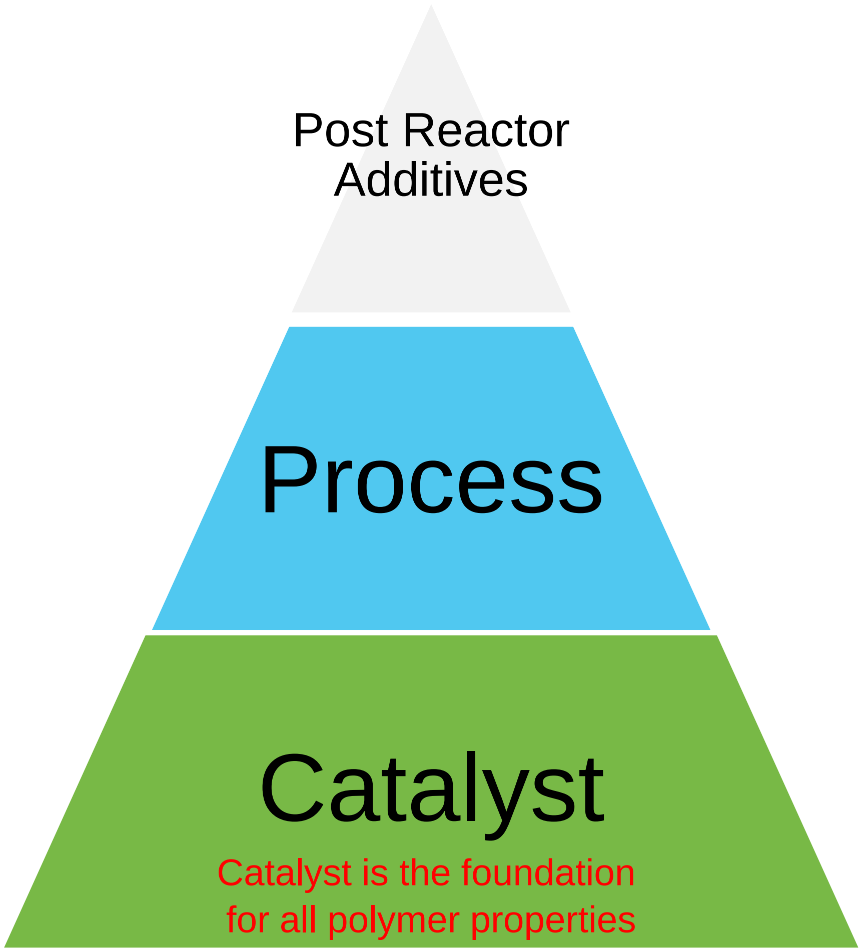
From Catalyst to Polymer



Catalyst Characteristics	Polymer Characteristics
Activity	Ash content, color (yellowness)
Isotacticity	Crystallinity, stiffness / softness
Hydrogen response	Molecular weight range (melt flow range)
Molecular Weight Distribution (MWD)	Stiffness, Processability
Surface Area and Porosity	Comonomer incorporation
Catalyst morphology (size and shape)	Process operability Comonomer incorporation

Catalyst Technology – Process Requirements

- Traditional requirements
  - High activity
  - High isotacticity
  - Low fines

- New (additional) requirements
  - Catalyst consistency
  - Good morphology
  - High polymer bulk density (for high throughput)
  - Very low micro fines
  - Stable operation

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Catalyst Technology – Process Requirements

Catalyst Property	Slurry	Bulk	Gas Phase
Catalyst activity	High	High	Medium
Particle Size	Small	M - L	Medium
Fines tolerance	Very low	Low	Low
PSD	Narrow	---	Narrow

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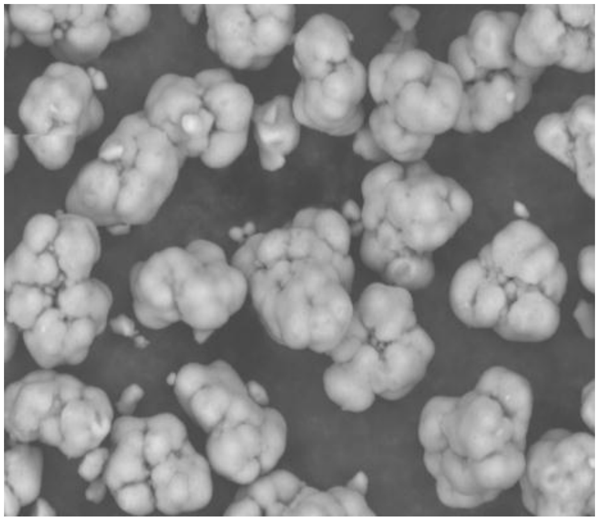
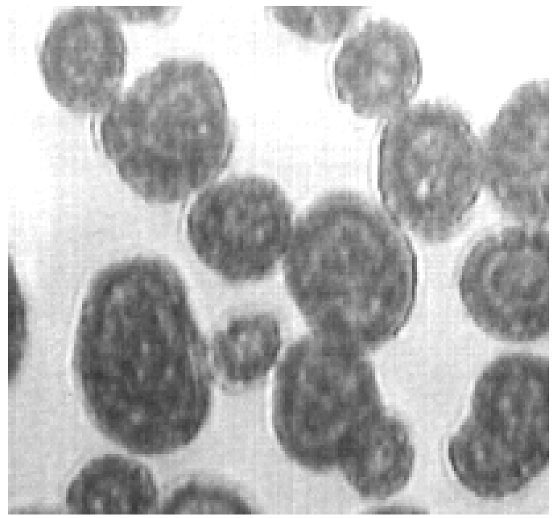
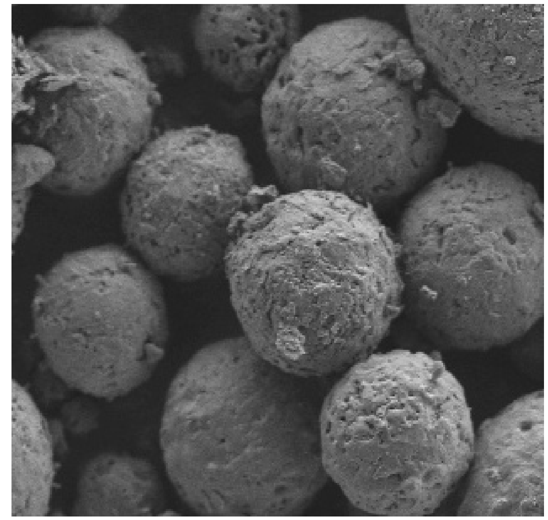
Catalyst Morphology







Granular

Spheroid

Spherical

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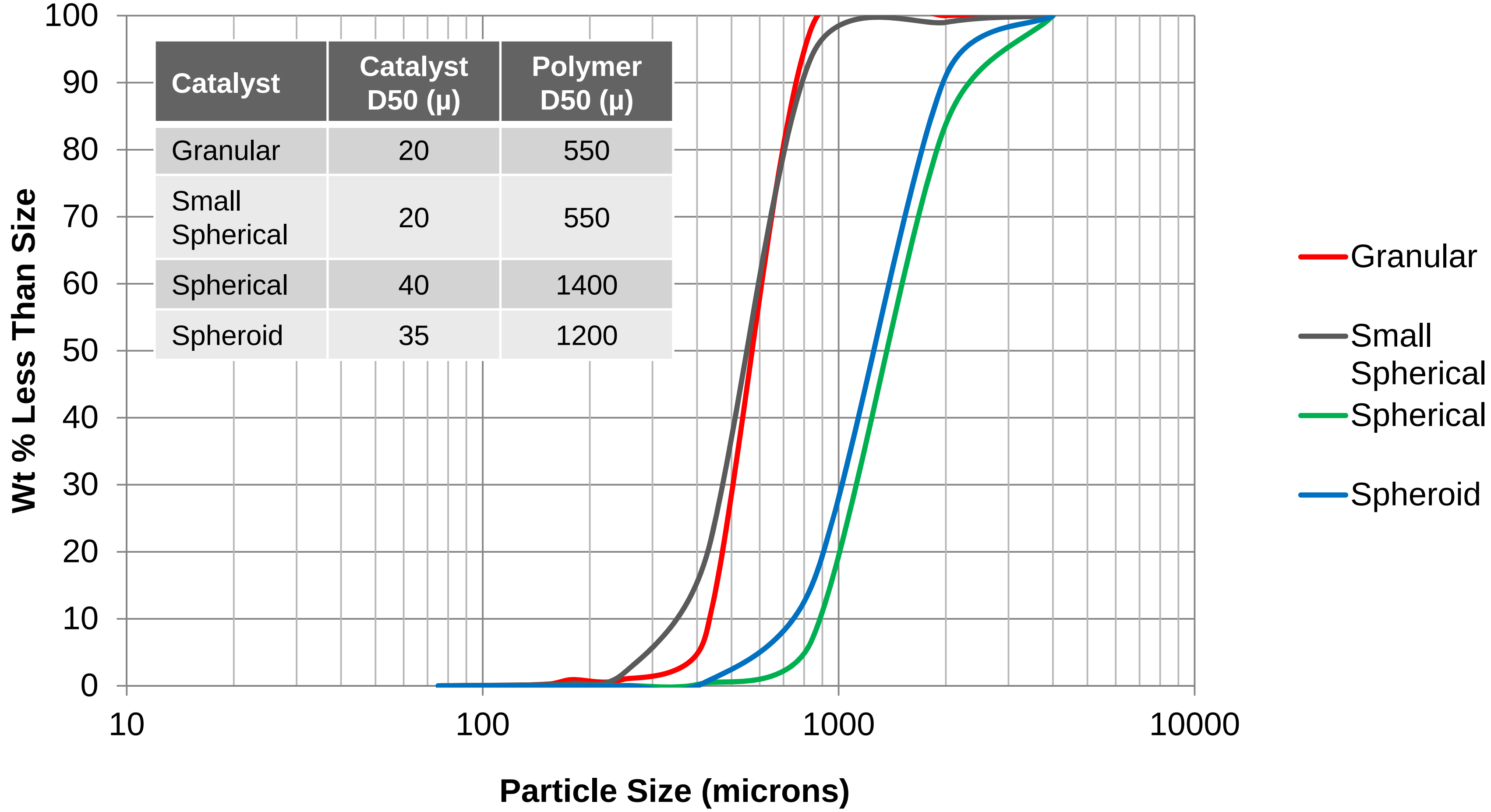
Polymer PSD from State of the Art Catalysts



Catalyst	Catalyst D50 (μ)	Polymer D50 (μ)
Granular	20	550
Small Spherical	20	550
Spherical	40	1400
Spheroid	35	1200

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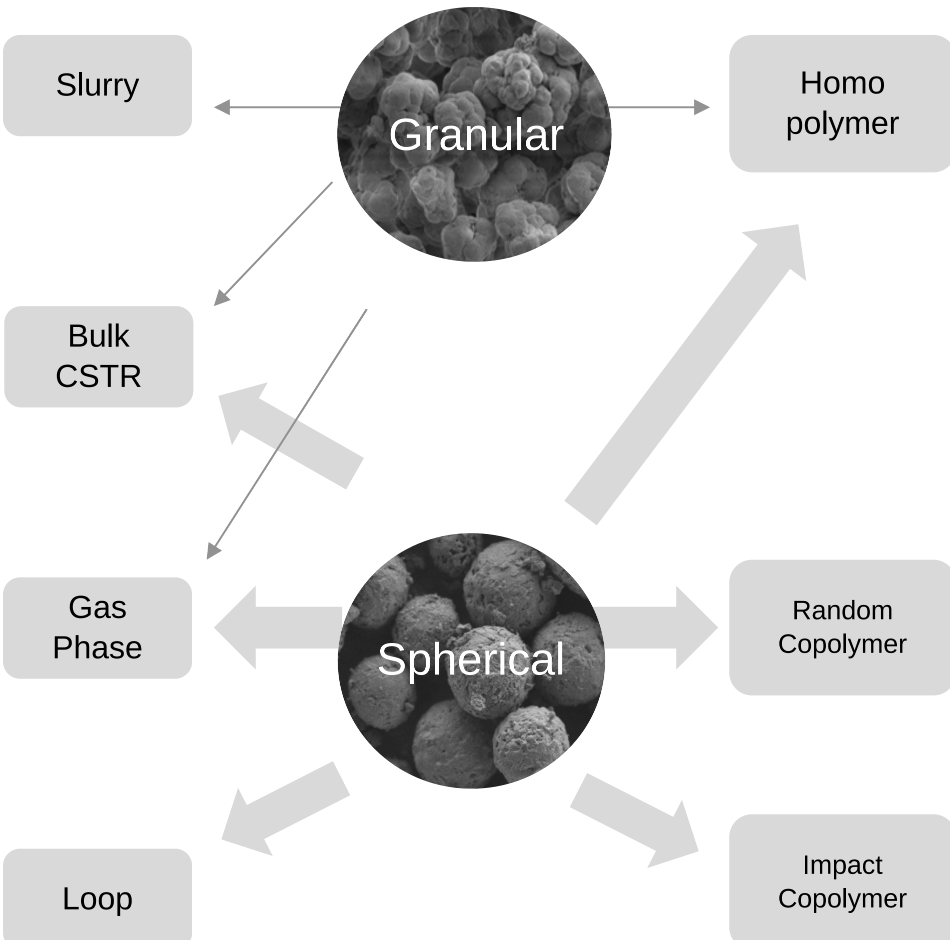
Catalyst Types for Different PP Processes and Products



Granular	Spherical
Limited particle size range (10 – 30 μ) Limited application in Loop process	Wide particle size range (15 – 80 μ) Applicable in all PP processes
Narrow PSD	Broad PSD
Less flowability due to irregular surface and particle shape	Better polymer flowability due to smooth surface and spherical shape
Mostly micropores, no macropores	Presence of macropores, suitable for impact copolymer
Most suitable for homopolymer	Suitable for all product grades

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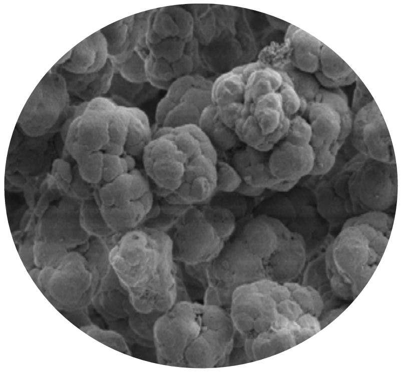
Why is Catalyst Morphology So Important?

– Most PP processes were originally designed around the licensor's catalyst with certain performance characteristics including
  - Polymer powder size – fluidization and powder transfer
  - Polymer powder shape –flowabilty and powder transfer
  - Polymer fines – operability issues including plugging of filters, sheeting, etc.
  - Polymer bulk density – reactor throughput and powder transfer



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Catalyst Technology – Product Requirements

Molding Applications

High Stiffness
High MRF, narrow MWD
Good impact-stiffness balance

Wide range of requirements

Medical

High purity
Low residue

BOPP

Broad MWD
Lower Stiffness

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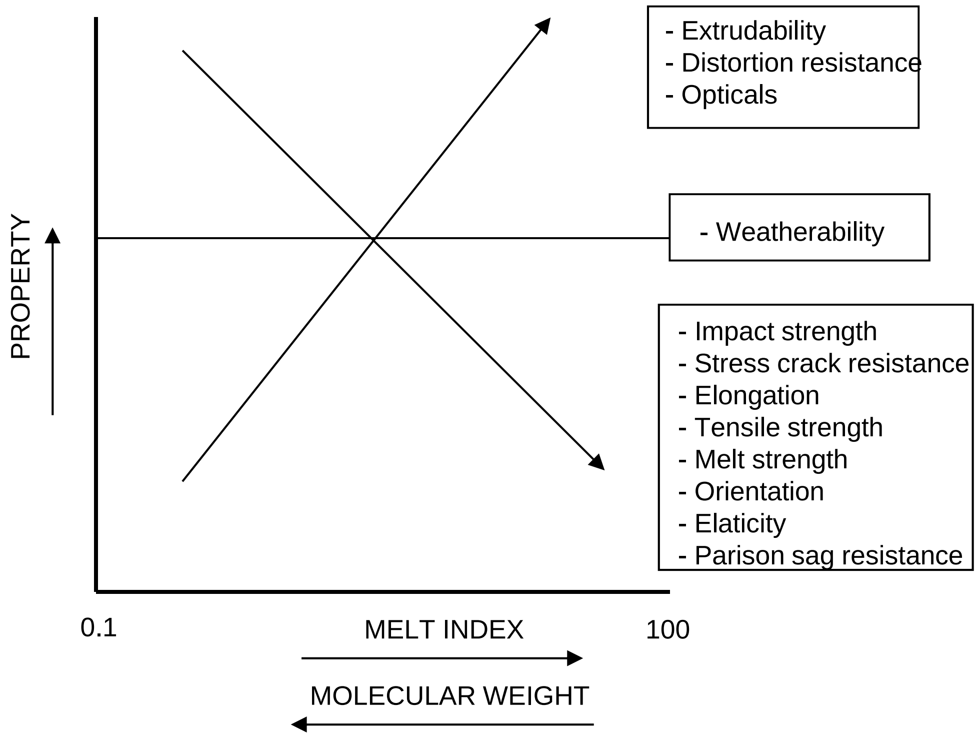
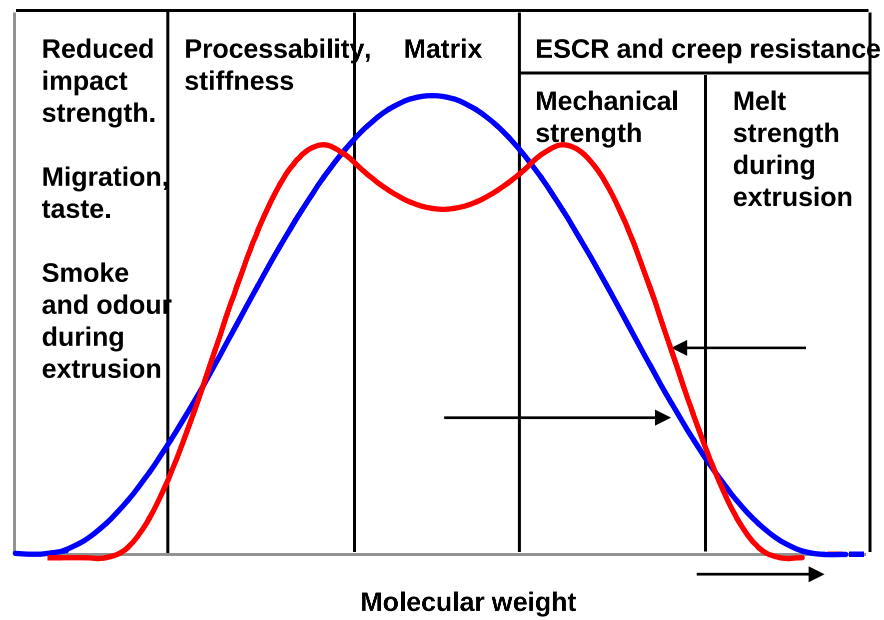
Polymer Properties





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Polymer Properties and Catalyst Properties

– Control of Molecular Weight Distribution
  - Improves stiffness
  - Improves polymer processability

– Hydrogen response
  - More push towards higher MF polymers for faster processing
  - Good hydrogen response need to achieve high MF

– Isotacticity
  - Controllable isotacticity is important for broad product portfoilio

– Comonomer response
  - High comonomer incorporation is desirable for high impact properties

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Polypropylene Applications

Conclusions and Future Trends

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Catalyst Development Trend

– Current 4th generation Z-N catalyst is already highly optimized
  - Basic recipe was developed almost 30 years ago
  - Most cost effective catalyst system for most PP products
– Most development efforts have been focused on

Mostly retained as trade secret	Many published in patent literature

- Further optimization of production recipe for incremental improvements in activity
- Improvements in catalyst morphology (size and shape control) for better process operability
	- Internal donor development
	  - Polymer properties enhancement
	  - Regulatory issues
	- External donor development
	  - Polymer properties enhancement

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Process Development Trends

- Gas phase process has gained significantly, especially coal-based PP plant in China
- Gas phase process is efficient for producing a small number of grades with high throughput

- Optimization of residence time distribution for more product homogeneity
- Optimization for better impact copolymer – higher rubber content, better ERP distribution etc.
- Use of Advanced Process Control (APC) for more stable operation and grade transitions

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Product Development Trends

– Impact copolymer
  - High MFR and high impact/stiffness balance for automotive applications – bumper, dashboard, under the hood applications
– Random copolymer
  - Medical grades
  - High end pipe grades (large diameter pipes)
  - High transparency random copolymer
– Terpolymer
  - Heat sealing layer in films
– Homopolymer
  - High MFR (melt blown) fiber
  - Capacity films
  - Extrusion coating
  - PP foam (HMSPP)

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Conclusions

- Catalyst, process and PP products are closely inter-related
- Development of one component often drive the development of the other components
- The growth of the PP industry mirrors the development of each generation of PP catalyst
- Current catalyst and production processes are already highly optimized with high efficiency
- Future growth will most likely driven by PP demand and not by technology development (unless there is a major breakthrough in technology)
- Breakthrough technology can come from PP product, catalyst, and process

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Thanks for Your Attention.

CLARIANT INTERNATIONAL LTD
Rothausstrasse 61
4132 Muttenz
Switzerland

CATALYSTS
petrochemicals@clariant.com

CLARIANT.COM/CATALYSTS

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