

Optimized Extrusion Process for developing High performance TPOs & lightweight Polyolefin Composites.

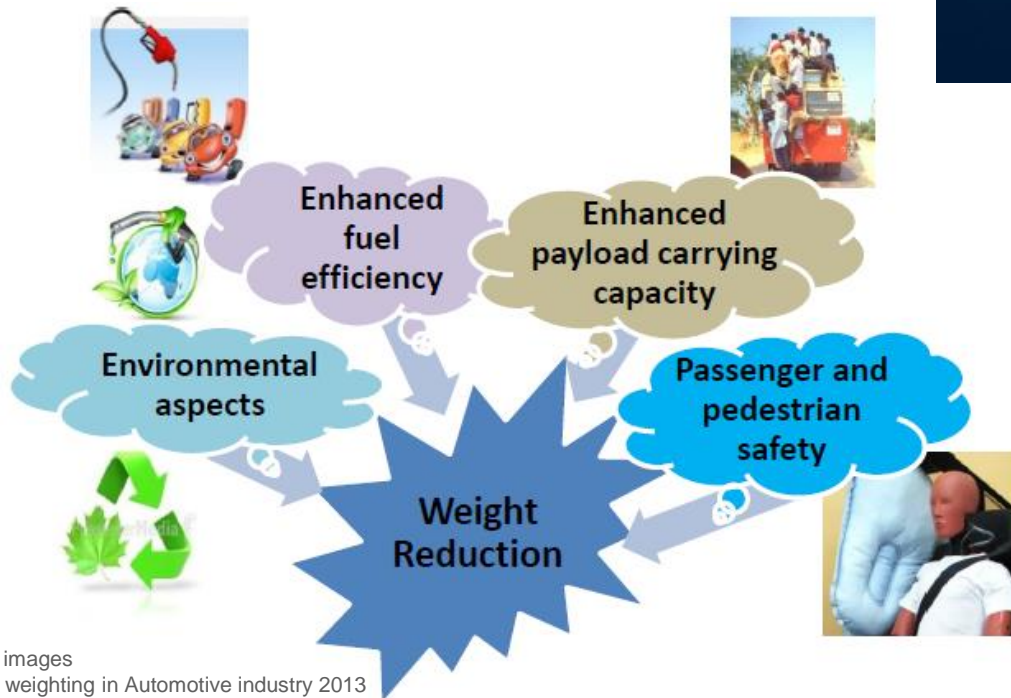
Dr. Tanmay J Pathak

Mr. Charles D Park II

Agenda

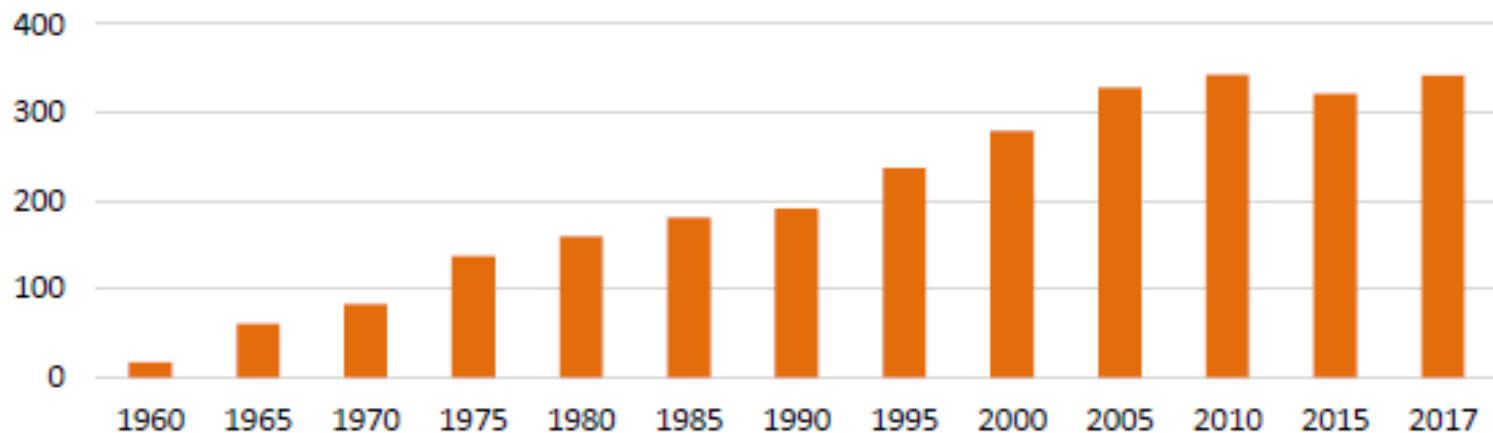
- **Light weighting – Importance in Automotive Industry**
- **Polymer resins and composites in Light Vehicles**
 - Historical Trend
 - Average Polymer type in light vehicles
- **Value of a Compounder – Property Balance**
 - Formulation Strategy & Process Selection
- **Twin Screw Extrusion**
 - Design Variables
 - Screw Design – Mixing
 - Distributive & Dispersive Mixing
- **Unique Process Design**
 - Mixing polymers with viscosity differentials – Unique Screw Design
- **Development of Light Weight Composite**
 - Density Reduction
 - Antagonistic Property balance

Light Weighting – Importance in Automotive Industry



(1) Ref: Shutterstock images
(2) Ref: ARAI – Light weighting in Automotive industry 2013

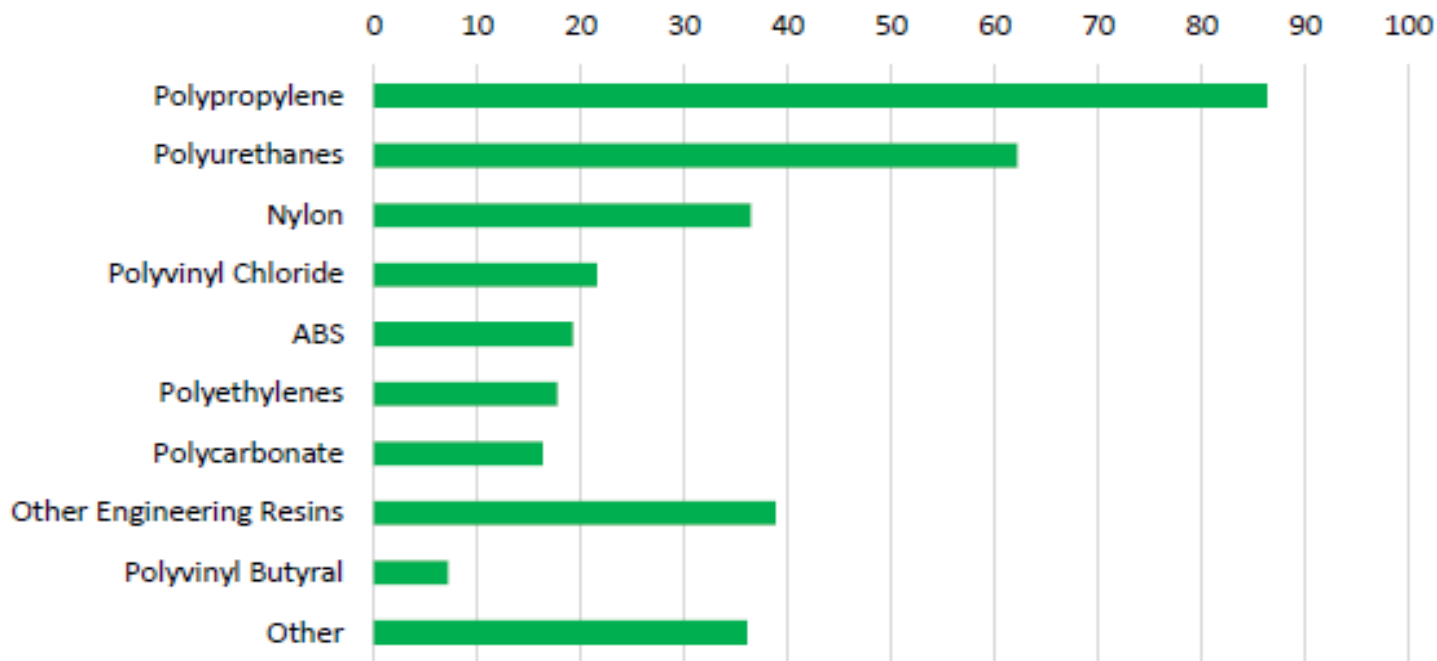
Polymers in Light Vehicles – Historical Trend



- Plastic components in vehicles have increased continuously over last 5 decades.
 - 1960 – average lbs. of plastic used < 20
 - 2017 – average lbs. of plastic used ~ 342
- Initial trend was to replace heavy metals with lighter plastic components
- Currently, already existing polymer composites are targeted to be replaced with even lighter composites

(1) Ref: Plastics & Polymer Composites in Light Vehicles, American Chemistry Council, (July 2018).

Polymers in Light Vehicles – Major Polymers used

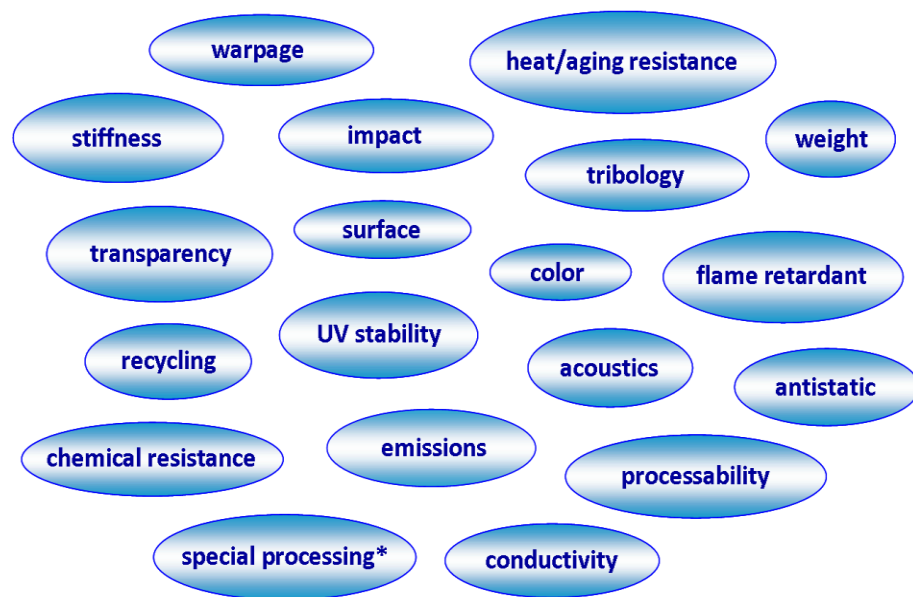


- **Polypropylene (PP) is a commodity polymer used widely in automotive and non-automotive industries.**
 - Average lbs. of PP used in light vehicles amounts to around 90 lbs. / vehicle
- **Engineered resins (inclusive of nylon, ABS, PC) when combined equal the usage of PP alone.**

(1) Ref: Plastics & Polymer Composites in Light Vehicles, American Chemistry Council, (July 2018).

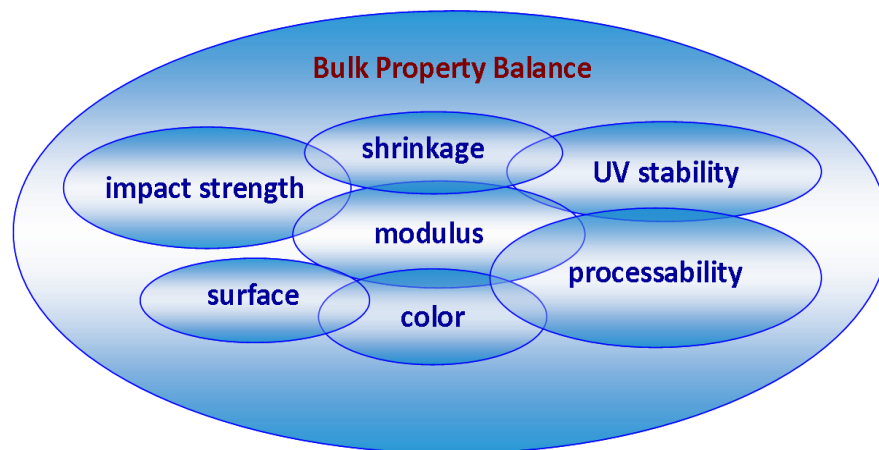
PROCESS DESIGN

Value of a Compounder – Property Balance

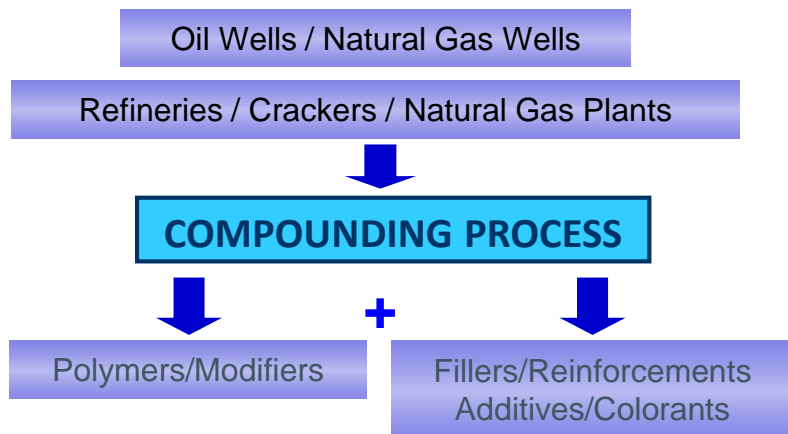


“the universe of performance”

Special value exists when more than one property is optimized!



Compounding Process



For modification of:

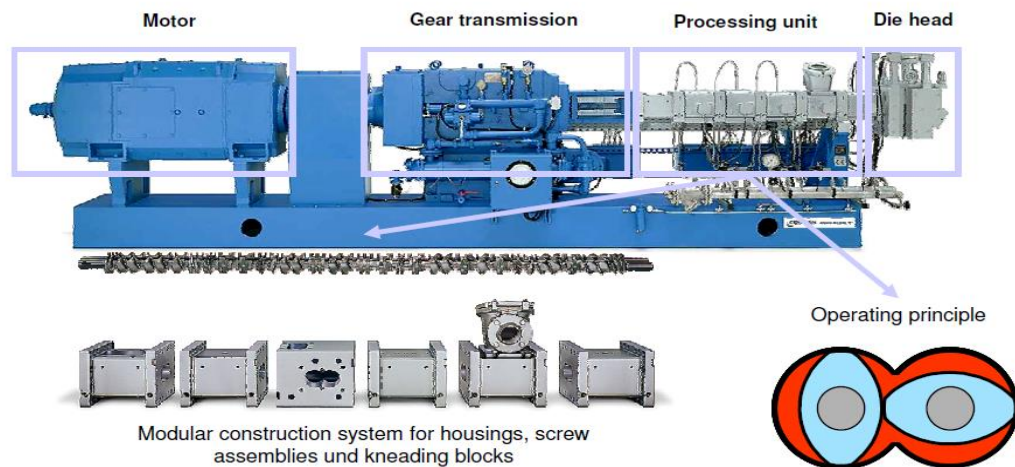
Material Properties

Appearance

Processability

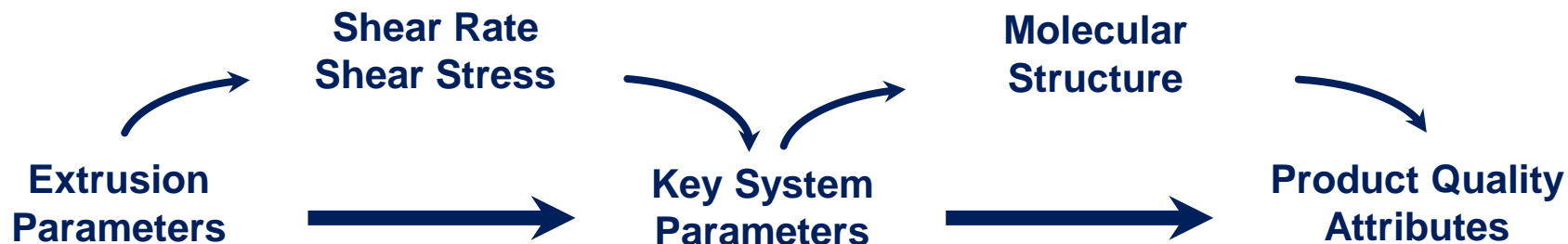
Polymers
Fillers/Reinforcements
Impact Modifiers
Lubricants
Surface Modifiers
Heat, UV, Other
Stabilizers
Plasticizers
Flame Retardants
Colorants

Twin Screw Compounding Extruder



(1) Data sources: LYB 2018

Factors associated with Twin Screw Extrusion (TSE)

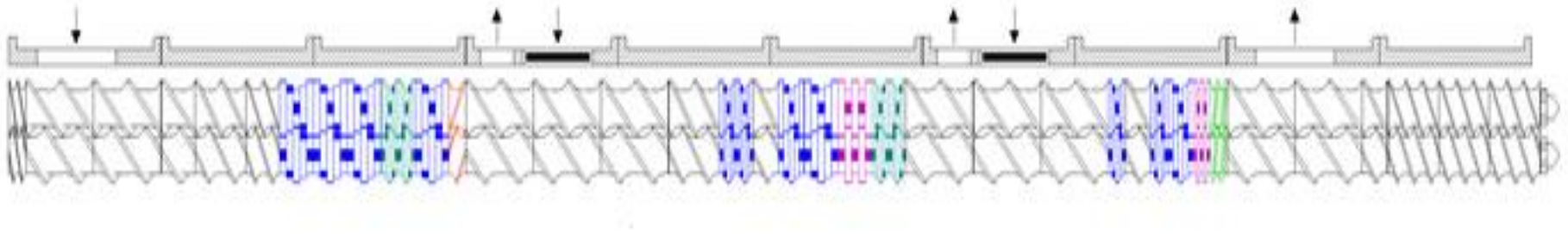


- ✓ Machine Parameters
 - Free Volume
 - Screw Configuration
 - Die Geometry
- ✓ Process Parameters
 - Screw Speed
 - Feed Rate
 - Barrel Temperature

- ✓ Specific Energy
 - Mechanical
 - Thermal
- ✓ Melt Temperature
- ✓ Residence Time
- ✓ Pressure

- ✓ Physical Properties
 - Morphology
 - Crystallinity
- ✓ Rheology
 - Mol. Weight
 - Mw Distribution
- ✓ Other
 - Color
 - Processability

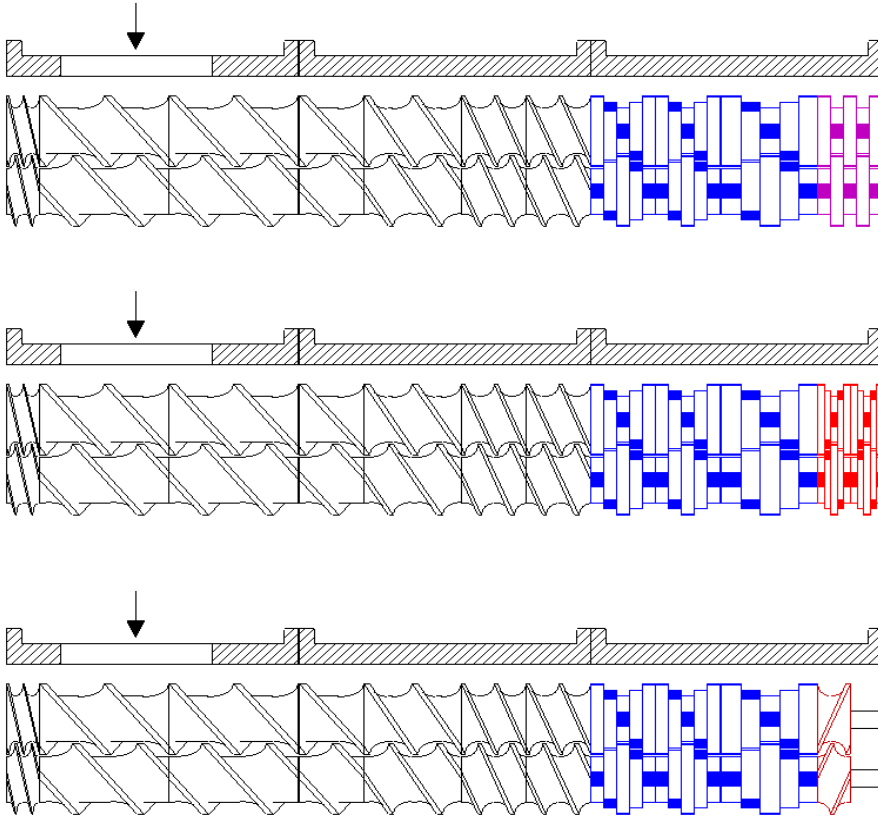
Twin Screw Extrusion – Screw Design



■ **A standard screw configuration involves assembly of elements on a screw shaft that allow**

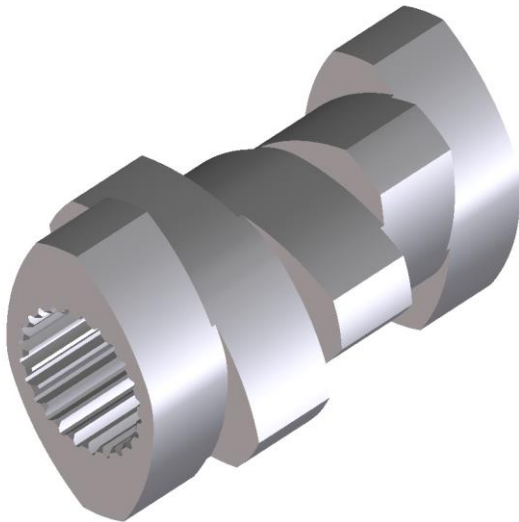
- Feeding - proper conveying of fed components (resins, fillers, additives), melting.
- Melting – Melting resins and additives before mixing
- Mixing – Distributive / Dispersive
- Venting – Strategic venting portals to allow discharge of unwanted components
- Pumping – efficient die pressurization so there is no back flow

Screw Design - Mixing



■ Kneading block geometry and orientation are important in creating effective mixing.

Mixing – Kneading Elements



■ Design variables for conveying screw elements

- Direction of conveying
- Offset Angle
- Pitch
- Number of paddles

Types of Mixing

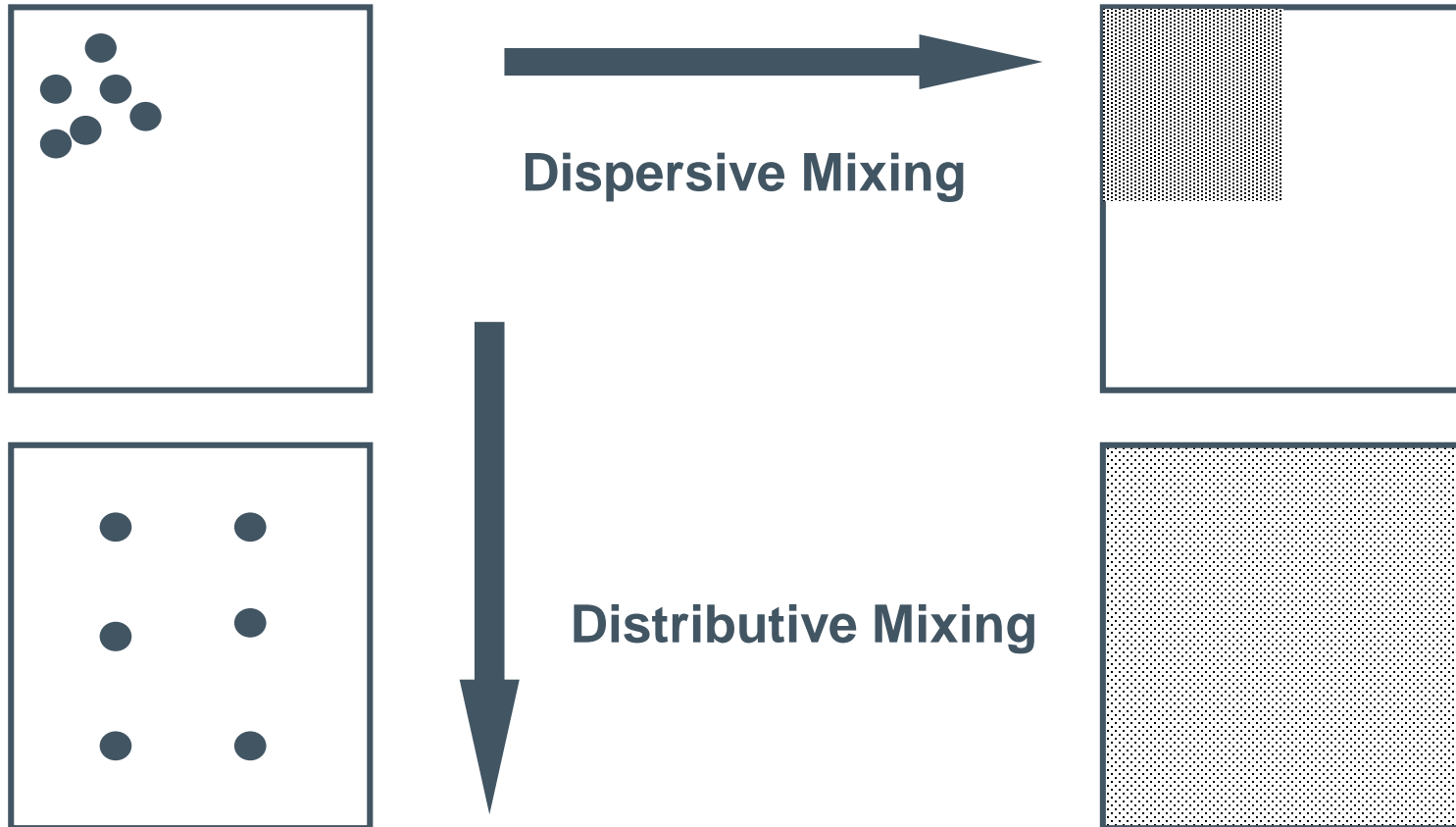
Distributive

- **Spatial rearrangement of species**
 - Distribution of fillers & additives
 - Thermal homogeneity
- **Involves reorientation / generation of new surfaces**
 - No reduction in size of domains

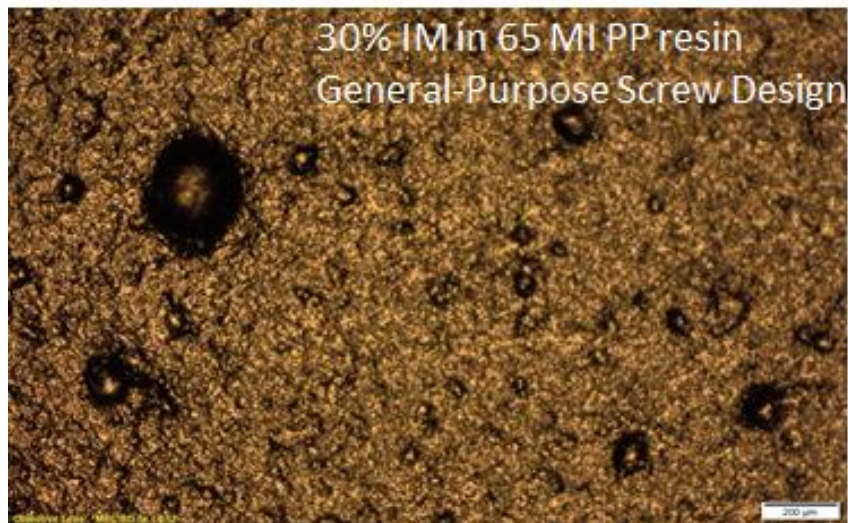
Dispersive

- **Reduction in size of domains**
 - Overcome shear stress which binds domains
 - $\text{Shear Stress} = \text{Shear Rate} \times \text{Velocity}$
- **Pressure – driven flow through clearances**
 - Spatial orientation not necessarily involved

Distributive v/s Dispersive



Unique Screw Design – Dispersion improvement

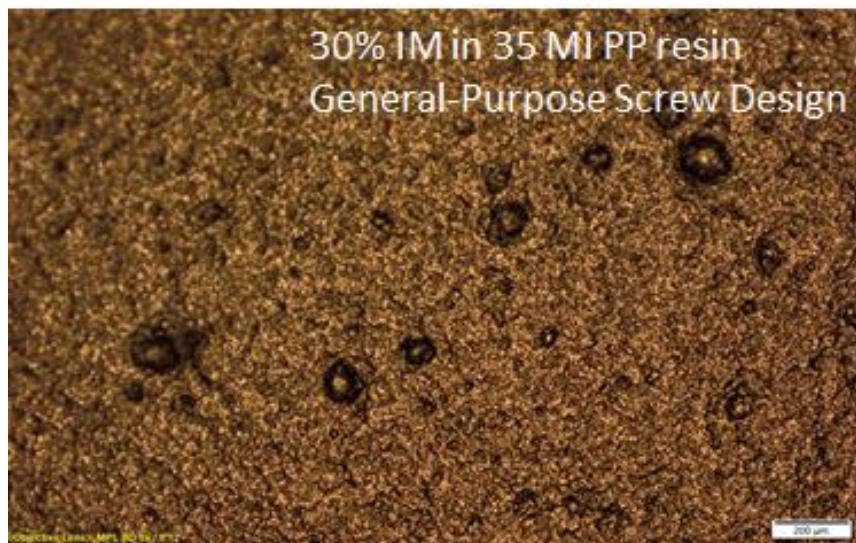


Low Melt Modifier in 65 MI PP

PROPERTY	GP Screw Design	Unique Screw Design
Melt Flow Rate (g/10min)	18	10
Flexural Modulus, Chord (MPa)	1038	864
Tensile @ Yield (MPa)	19	17
Elongation @ Break (%)	30	42
Notched Charpy Impact (kJ / m ²)	4	6
Multiaxial Impact [-30 ⁰ C] (J)	1.01	1.82

(1) Data sources: LYB 2018

Unique Screw Design – Dispersion improvement



Low Melt Modifier in 35 MI PP

PROPERTY	GP Screw Design	Unique Screw Design
Melt Flow Rate (g/10min)	13	8
Flexural Modulus, Chord (MPa)	1009	860
Tensile @ Yield (MPa)	20	17
Elongation @ Break (%)	58	81
Notched Charpy Impact (kJ / m ²)	6	10
Multiaxial Impact [-30 ⁰ C] (J)	1.70	15.60

(1) Data sources: LYB 2018

***Polyfort* PPC MT12 H1**

Light Weight Composite

- Development of the high-performance light weight composite was targeted towards a specific Automotive OEM requirement.
- A.Schulman (recently acquired by LyondellBasell) had an already approved 20% talc filled polypropylene composite to the OEM specification.
- OEM needed a lighter or reduced density product that would satisfy the same physical performance criteria as the 20% mineral filled composite.
- In addition to the physical performance, the new development had to pass all the long term weathering and heat ageing specifications.

OEM Specification

TARGET PROPERTIES

PROPERTY	Method	Units	Specification
Melt Flow Rate (230°C / 2.16kg)	ASTM D1238	g /10min	>20
Specific Gravity	ASTM D792	--	<1.00
Tensile @ Yield, 30 mm/min	ASTM D638	MPa	>21.6
Elongation @ Break, 30 mm/min	ASTM D638	%	>5
Flexural Strength, 30 mm/min, 0.25" thick	ASTM D790	MPa	>34.3
Flexural Modulus, 30 mm/min, 0.25" thick	ASTM D790	MPa	>2156
Notched Izod Impact @ +23°C	ASTM D256	J/m	>49
Notched Izod Impact @ -30°C	ASTM D256	J/m	>20
Modified DuPont Impact @ +23°C	OEM	J	>8.8
Modified DuPont Impact @ -30°C	OEM	J	>0.98
Heat Deflection Temperature, 0.45 MPa, 0.25" thick	ASTM D648	°C	>130
Heat Deflection Temperature, 1.80 MPa, 0.25" thick	ASTM D648	°C	>70
Rockwell	ASTM D785	R	>85
Gloss	ASTM D523	GU	<30

Materials

FILLERS for DOE

	D50 (micron)	Bulk Density (kg/m ³)
Filler 1	0.01	130
Filler 2	0.7	640
Filler 3	1.0	680
Filler 4	2.3	650

FILLERS for DOE

	Melt Flow Rate (230°C / 2.16kg)	Izod Impact (23°C)	Flexural modulus (1.3 mm/min)
CoPP	25 g/10min	No Break	1050 MPa
HPP	35 g/10min	26 J/m	1400 MPa

Imperative to identify the right combination of polypropylene resins, filler and additives.

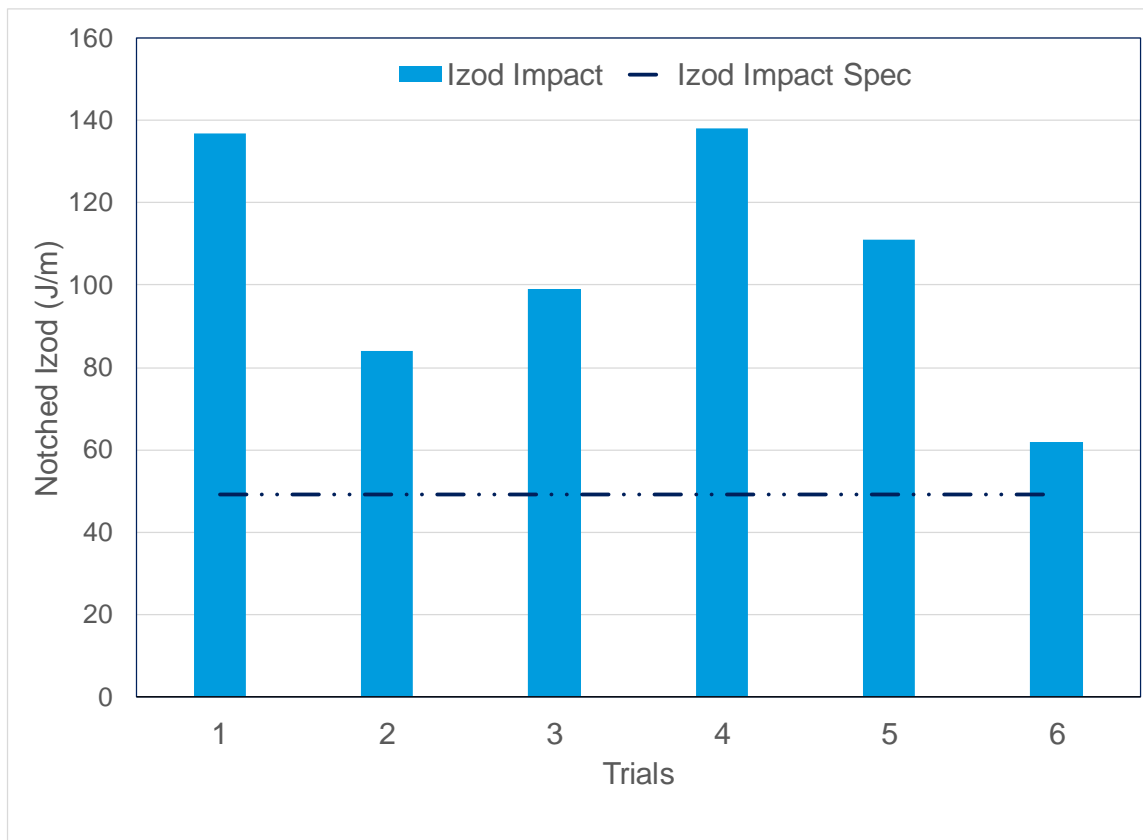
DOE 1

- The first set of trials were targeted towards finding the right balance of copolymer polypropylene (CoPP), homopolymer polypropylene (HPP) and filler.
- Two key properties that were kept in mind were stiffness (Flexural Modulus) and Impact (Izod), as it is well known that these properties are antagonistic to each other.
- Addition of high impact polymers can increase impact properties but tend to reduce the stiffness of the final product.
- Conversely, to achieve higher stiffness, impact has to be sacrificed.
- Higher stiffness can be achieved using higher filler levels but that can increase the density / specific gravity of the final product as well which was not desirable.
- Keeping this in mind, ONLY Filler 3 - with D50 = 1 micron was used to identify the combination of CoPP and HPP ratios that can get closest to target properties.

DOE 1 ran 6 trials on a general purpose screw design.

Impact Resistance – DOE 1

Target Impact: Notched Izod @ 23°C

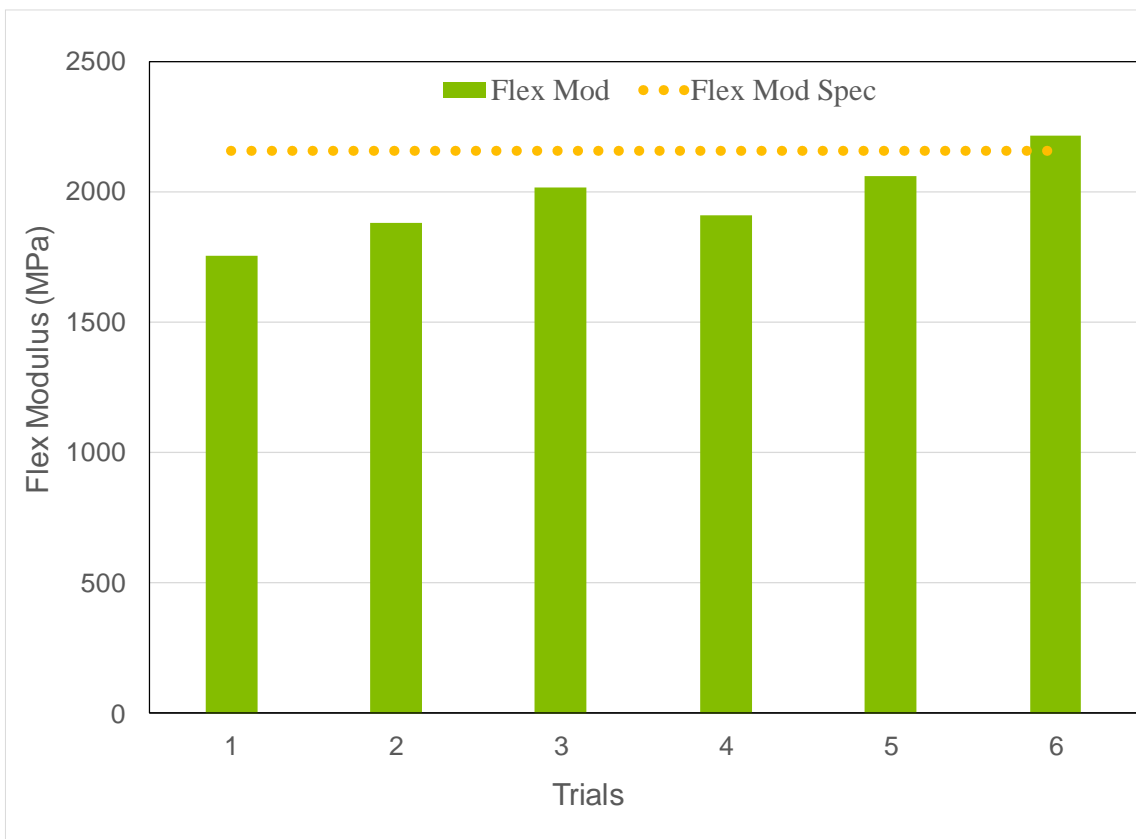


Given the combinations of CoPP and HPP on a GP screw design, all trials met the impact target

(1) Data sources: LYB – June 2017 .

Stiffness - DOE 1

Target Stiffness: Flexural Modulus

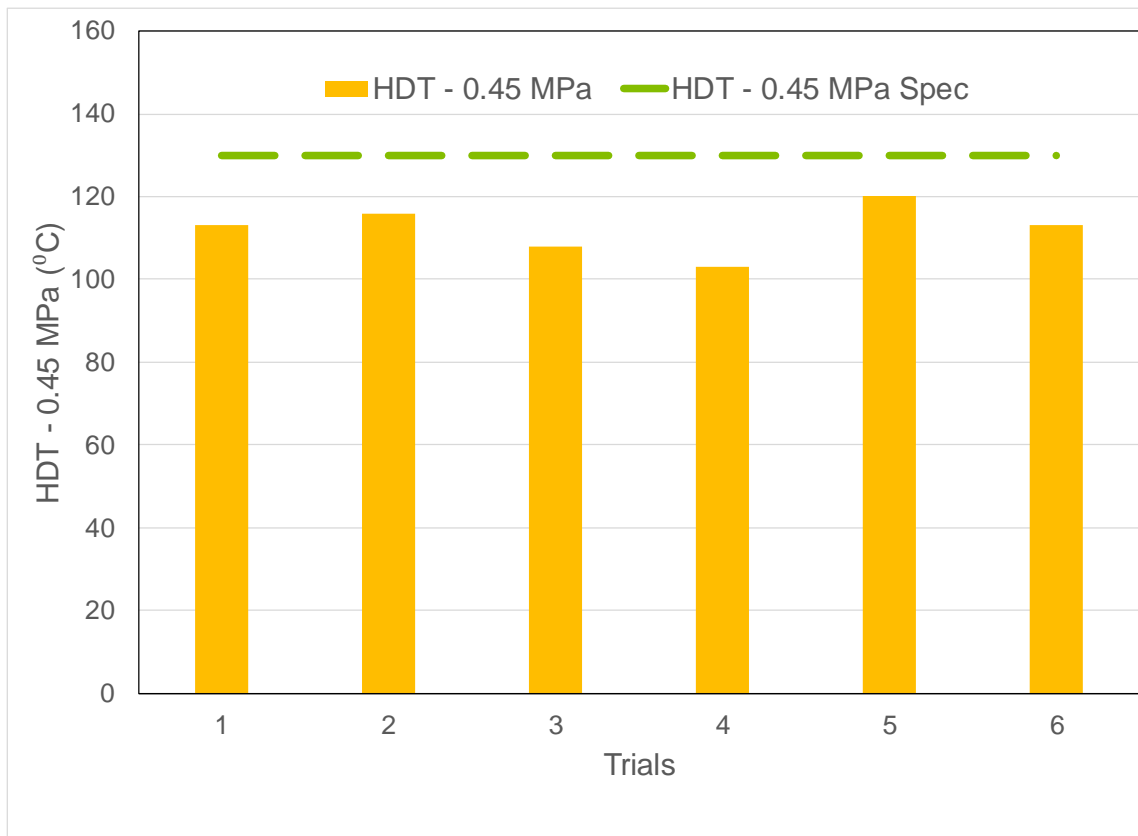


Given the combinations of CoPP and HPP on a GP screw design, only T6 met the stiffness target

(1) Data sources: LYB – June 2017 .

Heat Deflection Temperature - DOE 1

Target HDT: 0.45 MPa

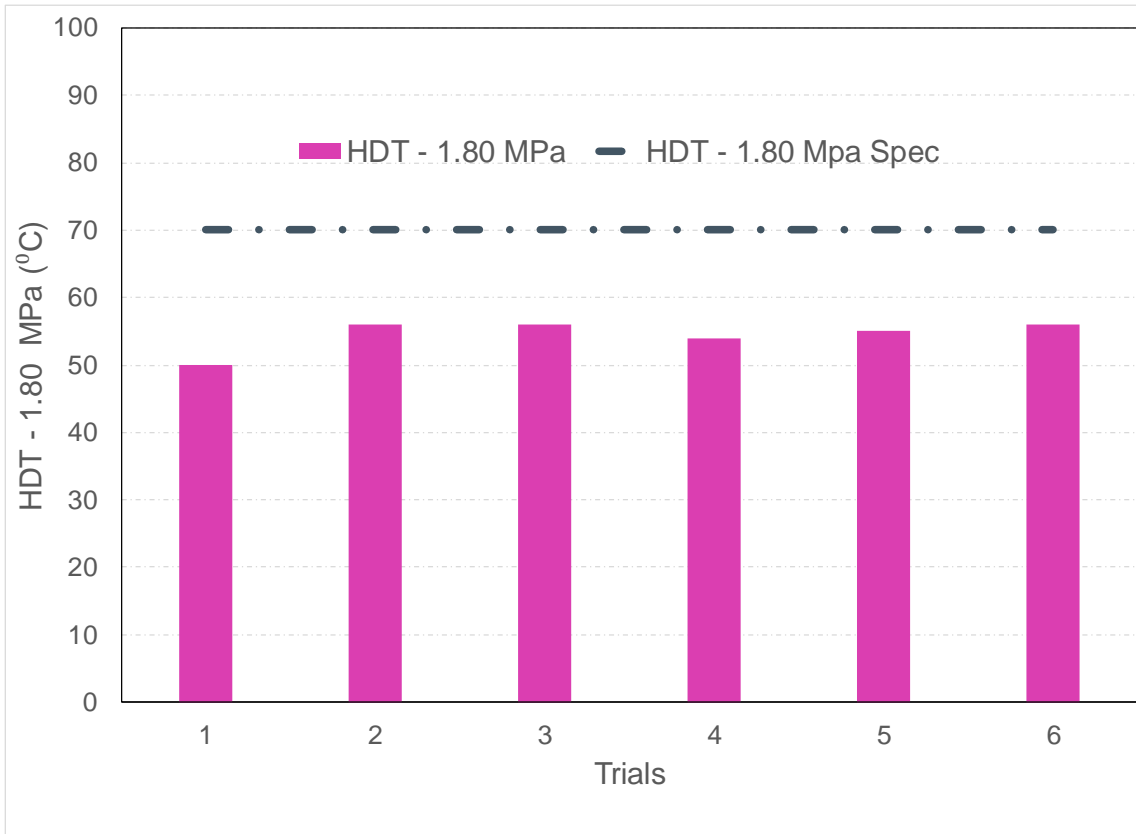


Given the combinations of CoPP and HPP on a GP screw design, No trials met the HDT at 0.45 MPa

(1) Data sources: LYB – June 2017 .

Heat Deflection Temperature - DOE 1

Target HDT: 1.80 MPa



Given the combinations of CoPP and HPP on a GP screw design, No trials met the HDT at 1.80 MPa

(1) Data sources: LYB – June 2017 .

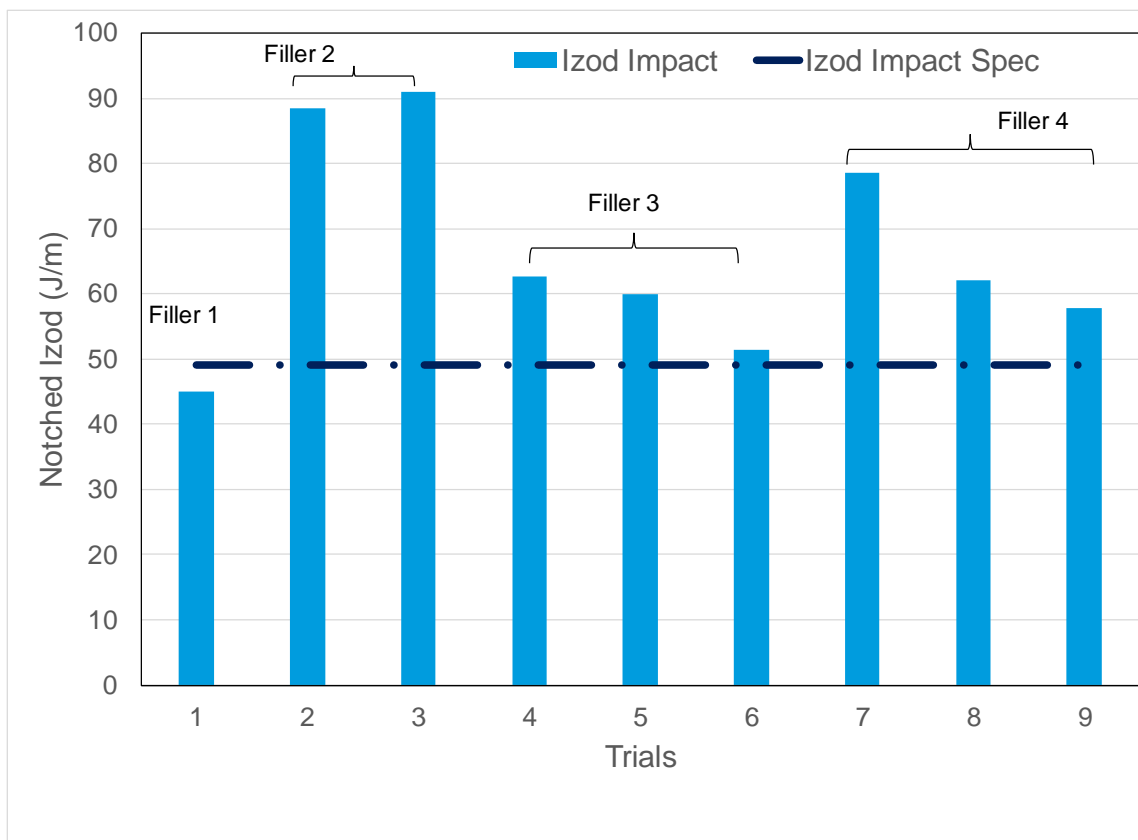
DOE 2

- DOE 1 gave an estimated resin ratio combination (T6) that was able to give a property balance between stiffness and impact.
- However, it seemed that the dispersion of the filler was not adequate to have higher stiffness properties.
- At the same time, a good filler dispersion is also necessary for creating more nucleating sites which can also improve the thermal properties like HDT
- As such, DOE 2 was designed to incorporate two aspects:
 - Different fillers with varying particle size and aspect ratios in the CoPP and HPP resin combination.
 - Unique LYB screw design that can enhance the dispersive mixing of a solid filler in high flow PP resin.
- DOE 2 focused on the same balance of stiffness and impact properties with the goal of increasing them well above the lower spec limit (LSL) and at the same time meeting all other properties such as HDT, hardness, gloss etc.

DOE 2 ran 9 trials on the unique LYB screw design.

Impact Resistance - DOE 2

Target Impact: Notched Izod @ 23°C

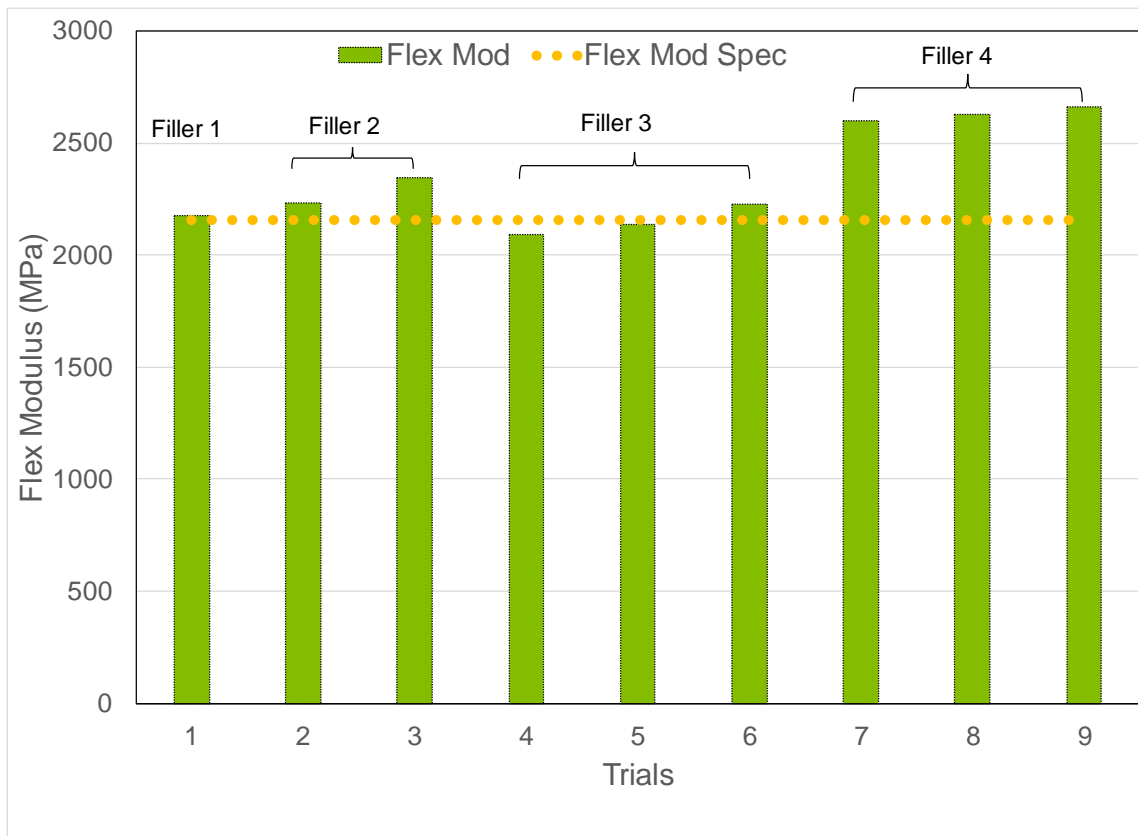


Given the combinations of CoPP and HPP on unique screw design, Filler 2 (T2, T3) and Filler 4 (T7, T8, T9) trials showed the best results.

(1) Data sources: LYB – February 2017 .

Stiffness - DOE 2

Target Stiffness: Flexural Modulus

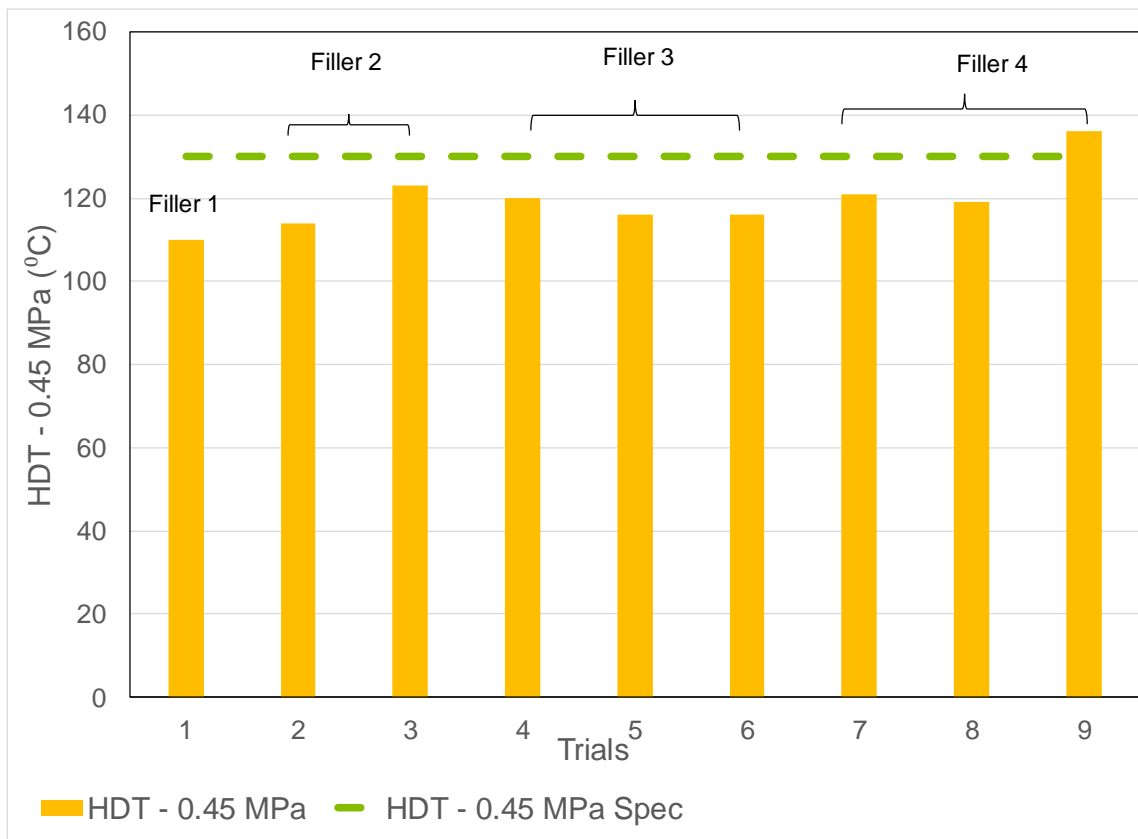


Given the combinations of CoPP and HPP on unique screw design, especially Filler 4 (T7, T8, T9) trials showed the best results.

(1) Data sources: LYB – February 2017 .

Heat Deflection Temperature - DOE 2

Target HDT: 0.45 MPa

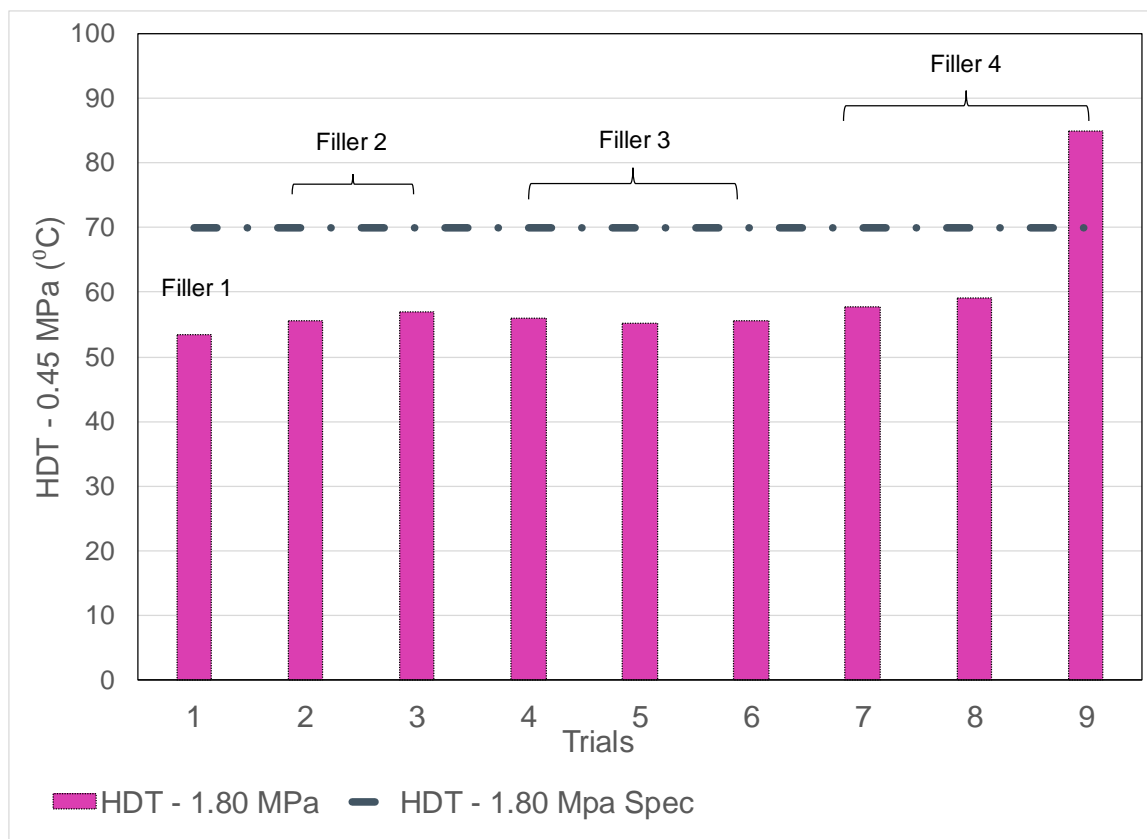


Given the combinations of CoPP and HPP on unique screw design, only T9 - Filler 4 met the HDT at 0.45 MPa

(1) Data sources: LYB – February 2017 .

Heat Deflection Temperature - DOE 2

Target HDT: 1.80 MPa



Given the combinations of CoPP and HPP on unique screw design, only T9 - Filler 4 met the HDT at 0.45 MPa

(1) Data sources: LYB – February 2017 .

OEM Specification – Lot Data

TARGET PROPERTIES

PROPERTY	Method	Units	Specification	Data
Melt Flow Rate (230°C / 2.16kg)	ASTM D1238	g /10min	>20	26
Specific Gravity	ASTM D792	--	<1.00	0.98
Tensile @ Yield, 30 mm/min	ASTM D638	MPa	>21.6	29
Elongation @ Break, 30 mm/min	ASTM D638	%	>5	16
Flexural Strength, 30 mm/min, 0.25" thick	ASTM D790	MPa	>34.3	48
Flexural Modulus, 30 mm/min, 0.25" thick	ASTM D790	MPa	>2156	2500
Notched Izod Impact @ +23°C	ASTM D256	J/m	>49	84
Notched Izod Impact @ -30°C	ASTM D256	J/m	>20	38
Modified DuPont Impact @ +23°C	OEM	J	>8.8	10.5
Modified DuPont Impact @ -30°C	OEM	J	>0.98	1.80
Heat Deflection Temperature, 0.45 MPa, 0.25"	ASTM D648	°C	>130	136
Heat Deflection Temperature, 1.80 MPa, 0.25"	ASTM D648	°C	>70	85
Rockwell	ASTM D785	R	>85	99
Gloss	ASTM D523	GU	<30	18

(1) Data sources: LYB – 2018.

THANK YOU!!

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Appendix

OEM Specification – Lot Data

TARGET PROPERTIES

Property	Test Method	Units	Spec	Data
Light Resistance (Xenon) - Izod Impact Retention after 90 MJ	ASTM D256	%	> 70	95
Light Resistance (Xenon) - Izod Impact Retention after 150 MJ	ASTM D256	%	> 60	96
Light Resistance (Xenon) - Tensile Yield Strength Retention after 90 MJ	ASTM D638	%	Report	96
Light Resistance (Xenon) - Tensile Yield Strength Retention after 150 MJ	ASTM D638	%	Report	96
Light Resistance (Xenon) –Elongation @ Break Retention after 90 MJ	ASTM D638	%	Report	93
Light Resistance (Xenon) –Elongation @ Break Retention after 150 MJ	ASTM D638	%	Report	72
Light Resistance (Xenon) – General Appearance after 90 MJ	AATCC-1	Visual	No Defects	No Defects
Light Resistance (Xenon) – General Appearance after 150 MJ	AATCC-1	Visual	No Defects	No Defects
Light Resistance (Xenon) – Color Retention after 90 MJ	ASTM D2244	DE	< 1.5	0.41
Light Resistance (Xenon) – Color Retention after 150 MJ	ASTM D2244	DE	< 1.5	0.45
Light Resistance (Xenon) – Gloss Retention after 90 MJ	ASTM D523	%	Report	97
Light Resistance (Xenon) – Gloss Retention after 150 MJ	ASTM D523	%	Report	95

(1) Data sources: LYB – 2018 .

OEM Specification – Lot Data

TARGET PROPERTIES

Property	Test Method	Units	IN-2 Spec	M100319
Flammability Initial	OEM	mm/min	≤ 81	49
Flammability Heat – 50 hrs	OEM	mm/min	≤ 81	66
Flammability Heat – 100 hrs	OEM	mm/min	≤ 81	58
Flammability Humidity – 50 hrs	OEM	mm/min	≤ 81	50
Flammability Humidity – 100 hrs	OEM	mm/min	≤ 81	72
Flammability Humidity – 336 hrs	OEM	mm/min	≤ 81	32

(1) Data sources: LYB – 2018 .